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ANALYTICAL ABSTRACTS

1.—GENERAL ANALYTICAL CHEMISTRY

529. **Use of mathematical statistics in analytical chemistry.** S. A. Gusinskaya (*Zh. Anal. Khim., SSSR*, 1954, **9** [4], 245–247).—The paper of Komar' (*Zh. Anal. Khim.*, 1952, **7**, 325) is discussed and some suggestions are made for shortening calculations. G. S. SMITH

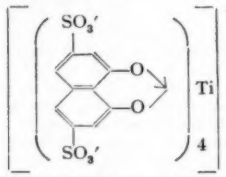
530. **Use of isotopes in analytical chemistry.** G. Heimann (*Chem. Rundschau*, 1954, **7** [18], 339–340).—The application of isotopes in analytical chemistry is discussed. The Geiger - Müller counter is described; an instrumental development from it, capable of registering correctly shaped and irregular impulses, is shown and explained. H. WREN

531. **Colorimetric reagents. II. Use of the oxime group.** Anon. (*Chem. Prod.*, 1954, **17** [10], 391–392).—The use of the metal derivatives of dioximes in colorimetric analysis is reviewed, *e.g.*, 1:2-*cyclo*-hexanedionedioxime yields a colloidal ppt. with Ni, easily stabilised with gum arabic and forming quantitatively at a lower pH than other reagents. Some turbidimetric and nephelometric methods are considered. G. R. WHALLEY

532. **Systematic examination of the uses of diethyldithiocarbamate in analysis. I.** H. Bode (*Z. anal. Chem.*, 1954, **143** [3], 182–194).—The spectra and dependance of distribution between aqueous phase and CCl_4 on the pH of the diethyldithiocarbamates of Ag^{+}, $As^{\cdots}$, Au, $Bi^{\cdots}$, $Cd^{\cdot\cdot}$, $Cu^{\cdot\cdot}$, $Fe^{\cdots}$, $Hg^{\cdot\cdot}$, $In^{\cdots}$, $Mn^{\cdots}$, $Ni^{\cdot\cdot}$, Os, $Pb^{\cdot\cdot}$, $Pd^{\cdot\cdot}$, Pt, $Sb^{\cdots}$, $Se^{\cdots\cdot}$, $Sn^{\cdots\cdot}$, $Te^{\cdots\cdot}$, $Tl^{\cdot}$, $Tl^{\cdots}$, $V^{\cdots\cdot\cdot}$ and $Zn^{\cdot\cdot}$ are given. The effects of the presence of the complexone and KCN on the distribution of the above metals, and of the ratio of concentration of metal to diethyldithiocarbamate are also shown. A convenient determination of Bi is possible; only $Tl^{\cdots}$, which can easily be reduced, interferes. If a spectrophotometer is available, Sb can also be estimated; As, Sn, Ge and Se do not interfere. A number of other possibilities including turbidimetric methods are suggested. (38 references.) P. S. STROSS

533. **Aromatic hydroxylamines as organo-analytical reagents.** G. D. Lutwick and D. E. Ryan (*Canad. J. Chem.*, 1954, **32** [10], 949–955).—Seven N-substituted phenylhydroxylamine compounds (N-*o*-ethoxybenzoyl-, m.p. 103° C; N-benzoyl-, m.p. 121° C; N-*o*-iodobenzoyl-, m.p. 128° C; N-naphthoyl-, m.p. 129° C; N-3:5-dinitrobenzoyl-, m.p. 133° C; N-furoyl-, m.p. 134° C; N-2:4-dichlorobenzoyl-, m.p. 137° C) and N-benzoylnaphthylhydroxylamine are prepared and tested as analytical precipitants. These compounds react similarly, increasing in selectivity with an increase in acidity of the oxime group. Thus in acid solution (1 per cent. or more) only V, Sn, Ti and Zr are precipitated. Substituents on the ring of the benzoyl group of N-benzoylphenylhydroxylamine, however, fail to improve on this reagent as a precipitant; the property of being able to precipitate Sn from a fairly acid medium is lost and increase in reagent acidity requires an increase in pH for complete pptn. of Cu. All the compounds specified can act as oxidants or reductants with an oxidation potential of $\simeq -1{\cdot}2$V. D. BAILEY

534. **Analytical evaluation of chromotropic acid.** A. Okáč and L. Sommer (*Coll. Czech. Chem. Commun.*, 1954, **19** [3], 477–492).—The reaction of chromotropic acid (1:8-dihydroxynaphthalene-3:6-disulphonic acid) with $Ti^{\cdots\cdot}$ in dil. and in conc. H_2SO_4 soln. is studied by spectrophotometric and potentiometric methods. In a soln. of pH 3·5 to 5, $Ti^{\cdots\cdot}$ reacts with the reagent to form a stable dissociated complex having λ max. at 470 mμ. The Job continuous-variation curves indicate that the compound has the formula—



In acetate buffer soln. of pH 5 to 7 and in citrate soln. of pH 2·1 to 2·4, an orange-yellow complex compound is formed; in citrate soln., the ratio of $Ti^{\cdots\cdot}$ to reagent is 1 to 2. The red-violet colour produced when $Ti^{\cdots\cdot}$ reacts with chromotropic acid in conc. H_2SO_4 is due to the formation of an oxidation product; a product with a similar absorption curve is obtained by the reaction of chromotropic acid with $K_2Cr_2O_7$ in 2 *N* H_2SO_4 or with $Cu^{\cdot\cdot}$ and $UO_2^{\cdot\cdot}$ in an ammoniacal medium. The red-violet colour corresponds to the consumption of one atom of oxygen per molecule of chromotropic acid; vigorous oxidation yields yellow products, the formation of which is accompanied by the consumption of 4 to 6 atoms of oxygen. With $Hg^{\cdot\cdot}$, chromotropic acid forms a non-homogeneous yellow ppt.; in analysis, the effect of $Hg^{\cdot\cdot}$ can be eliminated by converting it to undissociated compounds. The following method for the determination of Ti utilises the complex formed in dilute acid soln. *Procedure*—To 30 ml of the test soln. containing 0·1 to 13 μg of Ti per ml, add H_2SO_4, to make the acidity 0·1 *N*, and 2 to 5 ml of a freshly prepared 5 per cent. soln. of the disodium salt of chromotropic acid; add 20 to 25 ml of *M* Na acetate soln. to make the pH 3·5 to 5, and dilute to 100 ml with distilled water. After 20 min., measure the extinction at 470 mμ. The reagent should be present in ten-fold excess in both the test solution and in the standard. Chromate and ferric ions interfere; $Fe^{\cdots}$ is reduced by treatment of the soln. with hydroxylamine hydrochloride at 70° C. If present in high concn., HNO_3 must be removed by evaporating the sample with conc. H_2SO_4. The complex is also sensitive

to salts, and its colour is dependent on the ionic strength of the soln. [This is a translation into Russian of a paper previously published in *Chem. Listy*, 1953, **47**, 659.] E. HAYES

535. **Studies in oxidation - reduction reactions. II. Oxidation with chloramine-B. Indirect determinations.** Balwant Singh and Krishan Chander Sood (*Anal. Chim. Acta*, 1954, **11** [4], 317–323).—Substances that can be titrated with chloramine-B are used for a number of indirect determinations in which they are added in excess and the excess is then titrated according to the general procedure described earlier (*Anal. Abstr.* 1955, **2**, 261). In the following representations of these reactions the substance for determination appears first. In those reactions in which it is used arsenous oxide is dissolved in aq. NaOH. (*i*) $2H_3O_2 + As_2O_3 \rightarrow As_2O_5 + 2H_2O$. (*ii*) $PbO_2 + KI + 4HCl$ (in 4 *N* to 5 *N* HCl) $\rightarrow PbCl_2 + KCl + ICl + 2H_2O$. (*iii*) MnO_2 is treated in the same manner as PbO_2. (*iv*) $SeO_2 + N_2H_4.H_2SO_4 \rightarrow Se + N_2 + H_2SO_4 + 2H_2O$; the mixture is boiled and the Se removed by filtration. (*v*) $3Na_2S + As_2O_3 + 6HCl \rightarrow As_2S_3 + 6NaCl + 3H_2O$; the reaction mixture contains an excess of HCl; the As_2S_3 is filtered off. (*vi*) $4NaVO_3 + N_2H_4.H_2SO_4 + 12HCl$ (in 7 *N* to 7·5 *N* HCl) $\rightarrow 4VOCl_2 + 4NaCl + H_2SO_4 + 8H_2O + N_2$. (*vii*) $KIO_3 + As_2O_3$ (in 4 *N* to 5 *N* HCl) $\rightarrow As_2O_5 + KCl + H_2O + ICl$. (*viii*) $2CuSO_4 + 2KCNS + SO_2 + 2H_2O \rightarrow Cu_2(CNS)_2 + 2H_2SO_4 + K_2SO_4$; the soln. of $CuSO_4$ is saturated with SO_2, boiled with an excess of KCNS and the $Cu_2(CNS)_2$ is filtered off; the filtrate is boiled in an atmosphere of CO_2 to remove the SO_2. Sodium formate is treated with an excess of $HgCl_2$ and the soln., containing also Na acetate is heated on a steam-bath until no more CO_2 is evolved ($\simeq$ 2 hr.), $H{\cdot}COONa + 2HgCl_2 \rightarrow Hg_2Cl_2 + NaCl + HCl + CO_2$; the Hg_2Cl_2 is filtered off, transferred to a flask and titrated as usual. W. C. JOHNSON

536. **Micro-crystalloscopic determination of some rare elements.** V. D. Vasilenko, B. E. Reznik and E. E. Lutsenko (*Nauchn. Zap. Dnepropetrovskogo Gos. Un-ta*, 1953, **43**, 105–115; *Referativnyĭ Zh., Khim.*, 1954, Abstr. No. 16,804).—Quinoline can be used for the detection of In, Tl and Sb; 8-hydroxyquinoline for In, Sb, Sn and Tl; and dibromohydroxyquinoline for V, W, Mo, Ti, Tl, Sb, Sn and In. The last reagent can detect the elements in 4-component mixtures. The optical properties of the crystals formed, the sensitivity limits and the best conditions for carrying out the reactions are reported. E. HAYES

537. **Importance of polarisation curves in the application of electrochemical determinations.** R. Gauguin (*Chim. Anal.*, 1954, **36** [11], 285–290).—The mathematical properties of polarisation curves (intensity of electrolysis current against electrode potential) are presented. Necessary experimental conditions are deduced for general electrochemical analytical methods; these include amperometry at constant voltage and potentiometry at constant intensity, both with or without reference electrodes. D. R. GLASSON

538. **Instrumental methods of analysis.** W. van Tongeren (*Chem. Weekbl.*, 1954, **50** [49], 845–852).—The general principles underlying all types of instrumental analysis are discussed. E. HAYES

539. **Some uses of a galvanometer in analytical chemistry.** G. S. James (*S. Afr. Ind. Chem.*, 1954, **8** [8], 152–156).—The uses of a galvanometer and associated simple electrical equipment in various analytical methods, *e.g.*, oxidation - reduction titrations, polarographic methods, coulometric titrations, differential thermal analysis and turbidimetry, are described. These physico-chemical methods are recommended because they are rapid, accurate, sensitive and economical. G. C. JONES

540. **Use of oscillographic polarography in quantitative analysis.** R. Kalvoda and J. Mackŭ (*Chem. Listy*, 1954, **48** [3], 378–381).—A comparison method is described for a rapid quantitative determination of depolarisers by means of oscillographic polarography. Two curves of the function $d\bar{V}/dt = f_1(V)$ are obtained on the polarographic screen, one for the soln. of the depolariser under investigation and the other for the comparison soln.; the latter is then titrated with a standard soln. of the depolariser until both curves become identical and coincide. From the amount added, the concentration of the depolariser in the unknown soln. can be calculated. The error is usually $\not>$ 3 per cent. Details of the apparatus and circuit are given. G. GLASER

541. **Extension of the theory of the dead-stop titration.** J. H. Bradbury (*Trans. Faraday Soc.*, 1954, **50** [9], 959–965).—An extension to the theory of the dead-stop end-point titration, developed in an earlier paper (*Trans. Faraday Soc.*, 1953, **49**, 304), is given. A better quantitative development of the proposed theory is made by calculation of the ratio of the current between the electrodes to the diffusion constant in the Fichs diffusion equation (l/k_1) in the region of the equivalence point, as a function of volume of the titrant (V). A number of graphs of l/k_1 against V are shown, and, from these, the effect on the accuracy of the measurement at the equivalence point of altering various factors, from one titration to another, can be determined. Accurate titrations should be possible between redox systems whose standard potentials differ by only 0·200 V. Greater accuracy is obtained by working at low (*ca.* 0·010 V) rather than high values of the potential difference between the electrodes. Experimental results from the investigations of several systems are in fair agreement with theory. R. J. MAGEE

542. **The effect of oxide films and distance between electrodes on the precision of quantitative spectral analysis.** Josef Báláš (*Hutnické Listy*, 1953, **8** [7], 352–355; *Referativnyĭ Zh., Khim.*, 1954, Abstr. No. 20,347).—The results of a number of analyses show that the presence of oxide films on the electrodes and variations in the distance between the electrodes are material sources of error in spectral analysis. The effect of distance between the electrodes is most evident at low condenser [lens] apertures. It is recommended that a fully open condenser [lens] should be used and that a calibration curve should be made with the electrodes in the same condition as they are when used in the analysis. E. HAYES

543. **Horizontal alternating-current arc as an excitation source for the spectra of ores and minerals.** A. K. Rusanov and V. M. Alekseeva (*Zh. Anal. Khim., SSSR*, 1954, **9** [4], 183–192).—The advantages for spectrographic analysis of a horizontal carbon arc with a.c. are discussed. Spectra of parts of the flame along the axis of the electrodes and of parts along the length of the rising flame have

been examined. The results show a symmetrical distribution of intensities of arc and spark lines. Similar studies with d.c. arcs show a complex distribution of intensities. G. S. SMITH

544. **Polydimensional paper chromatography.** A. Stöckle (*Helv. Chim. Acta*, 1954, **37** [6], 1581–1585).—After preliminary unidimensional separation, the inadequately separated components are transferred in groups on to new sheets and further separated by specific solvents. Transference is effected by cutting out and stitching together the corresponding chromatogram strips. The process gives efficient separation even with mixtures rich in components. It requires only small vessels. A control can be run simultaneously with each experiment. The separation of twenty-two amino-acids by the new technique is described. H. WREN

545. **A new paper column for preparative chromatography.** L. Hagdahl and C. E. Danielson (*Nature*, 1954, **174**, 1062–1063).—The column described improves and simplifies an earlier chromatographic technique (Danielson, *Ark. Kemi*, 1953, **5**, 173). The filter-paper adsorbent is initially swollen by solvent so that it tightly packs the polythene-column tube and produces constant flow velocity throughout the cross-section, thus giving straight fronts and sharp separations for glycine, alanine and valine, for the glycyl deriv. of tryptophan, tyrosine and glycine in phosphate solutions and for metal ions. The amount of substances separated is limited only by the dimensions of the column. D. R. GLASSON

546. **Analysis of inorganic anions by paper chromatography. II. (Studies of analytical chemistry on filter-paper.)** S. Nakano (*J. Chem. Soc. Japan*, 1954, **75** [1], 71–73).—The paper-chromatographic distribution of a variety of anions (Na, K and ammonium salts, $\simeq$ 20 μg) has been examined with solvents containing 80 per cent. of ethanol and 20 per cent. of H_2O, *N* NaOH, *N* KOH, *M* Na acetate or 28 per cent. aq. NH_3 soln. The role of cations is closely examined and discussed. K. SAITO

See also Abstract 804.

2.—INORGANIC ANALYSIS

547. **Volumetric determination of water with Karl Fischer solution.** E. Eberius (*Chem. Rundschau*, 1954, **7** [18], 337–338).—An account is given of the nature, composition, manipulation and applications of the Karl Fischer solution. H. WREN

548. **An accurate method for determining water.** R. Leroy (*Chim. Anal.*, 1954, **36** [11], 294–301).—A mechanism for the dehydration of substances is presented. The amount of water lost by samples in atmospheres of different humidities can be calculated, when equilibrium is attained between the adsorbed water and the water vapour in the surrounding atmosphere. An equation for the curves of loss of water plotted against time is deduced. D. R. GLASSON

549. **Dichromate method of analysis of cations of analytical groups I, II and III.** S. M. Éfros and N. Z. Golynko (*Tr. Leningr. Tekhnol. In-ta*, 1953, [27], 76–102; *Referativnyĭ Zh., Khim.*, 1954, Abstr. No. 25,729).—A semi-micro method for the separation of group-II and group-III cations is based on differences in the solubility of the chromates. E. HAYES

550. **Determination of the lithium content of air.** I. I. Paul' and A. P. Vaĭsburd (*Gigiena i Sanitariya*, 1953, [9], 49–50; *Referativnyĭ Zh., Khim.*, 1954, Abstr. No. 16,789).—A semi-micro method for the determination of Li in air is based on its reaction with potassium periodate in the presence of $FeCl_3$ to form the insoluble $LiKFeIO_6$, which is dissolved in HCl; the iron is estimated colorimetrically with thiocyanate. The lower limit of sensitivity is 0·000245 mg of Li in 3 ml of soln. E. HAYES

551. **Determination of sodium as antimonate.** K. S. Cheshev (*Zh. Anal. Khim., SSSR*, 1954, **9** [4], 239–244).—Conditions for an accurate micro-method for determining Na as antimonate in the presence of large amounts of K are worked out. With K to Na ratios $>$ 50, pptn. of Na from a 24 to 25 per cent. ethanol medium is satisfactory, with K to Na ratios $>$ 100, the ethanol concn. should be 30 per cent., but with higher ratios excess of K is separated as KCl with ethanol. G. S. SMITH

552. **Papp's method for the determination of Na_2O content.** L. Zombory, M. Kenéz and M. Ferenczi (*Acta Chim. Hung.*, 1954, **4** [2–4], 181–194).—The electrodialytic method of Papp for the determination of the Na_2O content of red mud (*Aluminium*, 1950, **2**, 51) may also, with modifications, be used for the determination of the Na_2O content of red mud treated with CaO, or of artificial corundum, alumina or slags. N.E.

553. **Simultaneous determination of sodium, potassium, calcium, magnesium and strontium by a new multi-channel flame spectrometer.** B. L. Vallee (*Nature*, 1954, **174**, 1050–1051).—The new multi-channel flame spectrometer combines an oxy-hydrogen burner with a Wadsworth stigmatic mounting grating spectrograph; five photo-multiplier tubes, with power supplies for low and high voltage, and d.c. amplifiers are placed at the focal plane of the grating. Micro-ammeters, calibrated to read from 0 to 200 mA, record for each channel and provide 1 to 3 per cent. precision over a 1000-fold range of concn. Present limits of sensitivity in p.p.m. are: Na, 0·01; Ca, 0·1; K, 0·2; Mg, 0·3; Sr, 0·1. No internal standard is used. D. R. GLASSON

554. **Determination of sodium and potassium oxides by flame photometry in Portland-cement raw materials and mixtures and similar silicates.** C. L. Ford (*Anal. Chem.*, 1954, **26** [10], 1578–1581).—A finely ground sample of a silicate containing $>$ 100 p.p.m. of Na_2O or K_2O is ground with 0·5 to 1 g of NH_4Cl and 4 g of $CaCO_3$. The sample weight is chosen between 0·5 and 5 g, depending on the alkali content and on the Ca present in the silicate; low Ca contents require small sample weights. The mixture is compacted on 0·5 g of $CaCO_3$ contained in a 40-ml platinum crucible, 0·5 g of $CaCO_3$ being used to complete the transfer from the grinder. The crucible is heated to 900° to 1000° C during 1 hr. and this temp. is maintained for a further 1 hr. After cooling, the sintered cake is leached with hot water (100 ml) and the mixture is digested for $>$ 4 hr. The suspension is then boiled, filtered and, when cool, the filtrate is treated with 3 to 4 g of ammonium carbonate and the resulting suspension is filtered. This filtrate is evaporated to $\simeq$ 50 ml, neutralised with HCl (methyl red), cooled and made up to a standard vol. (so that K_2O or Na_2O concn. are $>$ 100

p.p.m.) in the presence of Ca soln. (112·5 g of $CaCO_3$ and 500 ml of conc. HCl per litre) (10 ml). The Na and K are determined flame-photometrically in this soln. by standard methods, the Ca being used as internal standard. Results are given for 21 silicate samples, and these agree well with those from gravimetric procedures. There is a time-saving of 50 per cent. D. A. PANTONY

555. **The simultaneous determination of potassium and triethanolamine by means of sodium tetraphenylboron.** R. Neu (*Z. anal. Chem.*, 1954, **143** [4], 254–257).—The method is based on the precipitation of both K and triethanolamine from solutions of their chlorides by means of a 3 per cent. solution of sodium tetraphenylboron. The precipitation should be carried out at 20° C at pH 5 to 6. After being kept for 24 hr. in a refrigerator, the solution is filtered and the precipitate, which consists of both the K and the triethanolamine salts of tetraphenylboron, is dried and weighed. The precipitate is then stirred with a mixture of ethanol (8 ml) and 70 per cent. $HClO_4$ (2 ml) for 1 hour and the suspension is filtered. The residue, $KClO_4$, is washed with ethanol (10 ml), dried and weighed. P. S. STROSS

556. **Determination of copper by precipitation with 2-(o-hydroxyphenyl)benzoxazole.** E. E. Byrn and J. H. Robertson (*Anal. Chem.*, 1954, **26** [10], 1605–1606).—A Cu^{++} soln., containing Fe^{+++} or Al^{+++} in small quantities only, is treated with a large excess of ethylenediaminetetra-acetic acid ($\simeq$ 5 g), and the pH is adjusted to 11 to 12 with 5 per cent. aq. NaOH soln. A slight excess of a saturated ethanolic soln. of 2-(*o*-hydroxyphenyl)benzoxazole, indicated by the development of a blue fluorescence in the soln., is added and the suspension is digested at 100° C for 15 to 30 min. The ppt. is filtered, washed with 50 per cent. ethanol and ether, and dried at 130° to 140° C. Interference from Fe^{+++} is avoided by the addition of a 30-fold excess of Na tartrate before pH adjustment, and that due to Al^{+++} by pptn. of the Cu^{++} at pH 3·5 to 4; the resulting ppt. is dissolved in conc. HCl and re-precipitated at pH 11 to 12 as above. Results of the analyses of Cu in a Cu - Ni alloy, a bronze, a cast iron and an aluminium alloy are given. Under the conditions described Ni^{++}, Mn^{++}, Co^{++}, Fe^{+++}, Cr^{+++}, Al^{+++}, Ti^{++++}, Zn^{++}, Sn^{++++}, Pb^{++} and Mg^{++} do not interfere. D. A. PANTONY

557. **Analysis of antifriction metals. Determination of copper. I. Rapid spectrophotometric method.** J. Hontoria, M. F. Gârate and M. T. Gârate (*An. Soc., Esp. Fis. Quim., B*, 1954, **50** [6], 549–552).—The metal, *e.g.*, white metal of low Cu content, is dissolved in HNO_3 - HF soln. The diluted solution is treated with aq. ammonium citrate and aq. NH_3 soln., followed by a solution of diethyldithiocarbamate. The coloured compound produced is extracted with butyl acetate and determined spectroscopically at 440 mμ. It is unnecessary to remove other metals. L. A. O'NEILL

558. **Studies on organic analytical reagents. VI. Conductimetric titration [of copper] with 8-hydroxyquinoline.** K. Shinra, K. Yoshikawa, T. Kato and Y. Nomizo (*J. Chem. Soc., Japan*, 1954, **75** [1], 46).—By making use of a newly designed conductimetric titrator with 6SN7GT vacuum tube, the volumetric analysis of Cu^{++} can be carried out with 0·02 *M* oxine soln. in 50 per cent. ethanol. The alkalimetric titration of oxine was also tried; the result was satisfactory with purified commercial oxine. K. SAITO

559. **Analysis of inorganic compounds by paper chromatography. I. The separation and detection of copper, nickel and cobalt.** S. N. Tewari (*Kolloidzschr.*, 1954, **133** [3], 178–181).—The separation of the sulphates of Cu, Ni and Co by upward, downward and horizontal chromatography is investigated on No. 1 Whatman paper. The effects on the R_F values of changes in solvent, temp., concn. of cation and movement of solvent front are given. Cu, Ni and Co give olive-green, violet-blue and yellowish-brown bands, respectively, after spraying with rubeanic acid in ethanol and exposure to NH_3. A. B. DENSHAM

560. **Simultaneous determination of copper, iron lead and zinc in copper-base alloys.** J. Kinnunen and B. Wennerstrand (*Metallurgia*, 1954, **50**, 149–150).—A method for the rapid analysis of brasses and bronzes is described. The elements Cu, Fe, Pb (plus Mn) and Zn are determined successively.

Procedure—A 0·5-g sample is dissolved in dil. (1 + 1) nitric acid (15 ml) with tartaric acid (3 g); after the addition of sulphamic acid (0·05 g), the solution is boiled to destroy brown fumes and cooled. Eight per cent. KI soln. (25 ml) or a soln. containing 5 g of KI and 60 g of NH_4CNS per litre (10 ml) is added and Cu is titrated with 4 per cent. w/v $Na_2S_2O_3$ soln. To determine Fe, boric acid (1 to 2 g) and dil. (1 + 1) nitric acid (10 ml) are added and the iodine liberated by Fe^{III} is titrated with $Na_2S_2O_3$ soln. After the addition of tartaric acid or mannitol (2 g), followed by ascorbic acid (0·2 g), 20 per cent. KCN soln. (15 ml) and conc. aq. NH_3 (20 ml), the solution is cooled; a manganese salt solution (5 to 10 ml) made by titrating 0·05 *N* $MnSO_4$ soln. with standard complexone III soln. until blue is added and Pb (plus Mn) is titrated with 0·01 *N* complexone III soln. Four per cent. formaldehyde soln. (20 ml) is then added and Zn is titrated with 0·05 *N* complexone III soln. Modifications are necessary when Fe or Al are present in appreciable quantities. If the Pb and Zn contents are low, extraction with sodium diethyldithiocarbamate is preferred; the procedure is outlined. G. C. JONES

561. **Chemical studies on radioactive indicators. XVIII. Controlled-potential electro-separation of copper, bismuth and lead and its radioactive indication by use of thorium-B and thorium-C tracers.** M. Ishibashi, T. Fujinaga and Y. Kusaka (*J. Chem. Soc. Japan*, 1954, **75** [1], 13–14).—When a solution containing Cu, Pb and Bi ions is electrolysed under the prescribed conditions, these metals can be successively deposited on the cathode and hence determined accurately (*Japan Analyst*, 1953, **2**, 342–345). *Procedure*—The sample solution (0·1 *M* succinic acid, 0·1 *M* KCl, 0·06 *M* hydrazine and 0·3 *M* tartaric acid, pH 5·9) is electrolysed at a cathode potential of −0·30 V *vs.* the S.C.E. for Cu (50 to 100 mg), then at −0·40 V *vs.* the S.C.E. for Bi (50 to 100 mg) and finally at −0·60 V *vs.* the S.C.E. for Pb ($\simeq$ 50 mg). This method was examined with the aid of thorium-B (for Pb) and thorium-C obtained from Ra - Th source (for Bi). The mutual separation of these elements was proved quite satisfactory although a small amount of Pb remains in the solution. K. SAITO

562. **Polarographic determination of gold in ruby glass.** J. Kvaček (*Československ. Sklář a Keramik*, 1953, **3** [10], 183–184; *Referativnýĭ Zh., Khim.*, 1954, Abstr. No. 22,148).—Fuse, in a muffle furnace, 1 g of the finely ground sample with 15 g of Pb (granulated), 0·4 g of Ag and 1 g of $Na_2B_4O_7$. Powder the

resulting alloy of Au, Ag and Pb, and remove the Pb by cupelling. Dissolve the Ag by boiling with 2 ml of HNO_3 (sp. gr. 1·2); wash the residual Au with distilled water and dissolve it in a few drops of a (1 + 3) mixture of conc. HNO_3 and HCl. Remove the HNO_3 by evaporation on a water-bath at 70°C, add a little distilled water and pour the soln. into a 10-ml calibrated flask containing ≃ 2 ml of 2 *N* KOH. Add 0·5 ml of gelatin soln. [50 ml of H_2O, 0·25 g of gelatin and 3 ml of dil. HCl (1 + 1)] and after 30 min. polarograph as described by Linhart (*Chem. Listy*, 1950, **44**, 159).

E. HAYES

563. **Determination of beryllium in bronzes by means of cation-exchange resin.** D. I. Ryabchikov and V. E. Bukhtiarov (*Zh. Anal. Khim., SSSR*, 1954, **9** [4], 196–198).—The oxalate complex of Be can be adsorbed by the cation-exchange resin cationite SBS at pH 4·4 but the oxalate complexes of Al and Fe cannot. To determine Be in bronze, 0·5 g of the alloy is dissolved in 25 ml of 30 per cent. nitric acid; copper is removed by electrolysis. The spent electrolyte at 70° C is treated with aq. NH_3 soln. to ppt. beryllium, aluminium and iron hydroxides, which are then filtered off, washed and dissolved in 20 per cent. HCl soln. The solution is evaporated to 1 ml, 25 ml of water and 40 ml of 2 per cent. oxalic acid solution are added and the solution is made just alkaline to methyl orange with aq. NH_3 soln. and then passed through a column of SBS (15 g in a 100-ml burette) at the rate of 2 ml per min. The column is washed with water until no reaction for Fe with KCNS is obtained. Beryllium is extracted from the column by using a 10 per cent. soln. of HCl at 4 to 5 ml per min. About 250 ml are required. Beryllium is pptd. as hydroxide, which is ignited to oxide and then weighed.

G. S. SMITH

564. **Detection of calcium according to Gutzeit.** Z. Řezáč (*Chem. Listy*, 1954, **48** [3], 471–472).—Gutzeit's method for the detection of Ca with an ammoniacal soln. of phenol (*Helv. Chim. Acta*, 1929, **12**, 829), alleged to produce a blue colour, has been found worthless; no reaction occurs. G. GLASER

565. **Determination of small amounts of calcium with pyrazole blue.** L. Erdey and L. Jankovits (*Acta Chim. Hung.*, 1954, **4** [2–4], 235–244).—Picrolonic acid is the best pptg. agent for the determination of Ca in alumina, but with minute quantities of the element the colorimetric measurement of pptd. Ca picrolonate is difficult owing to the low value of extinction; it is preferable to convert the picrolonic acid into pyrazole blue. *Procedure*—The neutral calcium solution in a 15 to 20-ml centrifuge tube is constantly stirred and treated gradually with 0·01 *N* picrolonic acid (3 ml) and centrifuged after 24 hr. The ppt. is washed with water saturated with Ca picrolonate (1 ml). The united solutions are treated with 2 *N* H_2SO_4 and ≃ 10 per cent. $K_2S_2O_8$ at 100° C for 5 to 10 min. and the pptd. pyrazole blue is extracted by chloroform from the cooled solution. The colour of the $CHCl_3$ solution is measured after 30 min. with a Pulfrich photometer and an S53 filter. Alternatively, the pptd. Ca picrolonate is dissolved in HCl soln. and oxidised to pyrazole blue, which is colorimetrically determined. H. WREN

566. **Application of bromanilic acid (3 : 6-dibromo-2 : 5-dihydroxy-*p*-benzoquinone) in analytical chemistry. Colorimetric determination of calcium with sodium bromanilate.** L. Erdey and L. Jankovits (*Acta Chim. Hung.*, 1954, **4** [2–4], 245–258.—To a neutral solution containing 0·1 to 0·2 mg of Ca^{++} in a 50-ml calibrated flask is added distilled water (up to 30 ml). Exactly 10 ml of 0·3 per cent. Na bromanilate are added, the flask is stoppered and the solution is well shaken. After remaining for 3 hr. at room temp., with shaking every half hour, the solution is made up to 50 ml with distilled water, the solution is well shaken and either centrifuged or passed through a hard ash-free filter-paper into a dry beaker. The clear filtrate is observed at 530 mμ in a Pulfrich photometer. Distilled water is used as a comparison solution. The calibration curve is prepared similarly with known amounts of a Ca solution. Sodium bromanilate appears unsuitable for the volumetric determination of Ca, but may be suitable for the gravimetric method. The preparation of Na bromanilate is described.

H. WREN

567. **Determination of small quantities of zinc in the air of industrial undertakings.** S. I. Gusev and Z. A. Bitovt (*Gigiena i Sanitariya*, 1953, [11], 48–50; *Referativnyĭ Zh., Khim.*, 1954, Abstr. No. 23,960).—A photo-nephelometric method for the determination of 0·12 to 1 μg of Zn in air is based on the reaction between Zn^{++} and diantipyrinylmethylmethane hydrochloride in the presence of CNS′ at pH 2 to 2·5. The turbidity is directly proportional to the Zn^{++} content of the soln. Zinc can be determined in the presence of concn. of Cu^{++} and Fe^{+++} several times greater than that of the Zn.

E. HAYES

568. **Determination of surface zinc oxide on zinc sulphide phosphors.** S. Larach and S. M. Thomsen (*Anal. Chem.*, 1954, **26** [10], 1600–1601).—To 10 ml of water containing 1 drop of methyl orange (0·1 per cent.) is added sufficient 0·1 *N* HCl (≃ 1 drop) to develop the pink colour of the indicator. The ZnS phosphor (0·5 to 1 g) is thoroughly stirred in; if there is no change of indicator colour, the ZnO concn. is < 0·02 per cent., but for appreciable ZnO concn., the titration with 0·1 *N* HCl is continued slowly until the pink colour is permanent. ZnO determinations on three synthetic samples are given as examples. D. A. PANTONY

569. **Iodimetric determination of cadmium.** V. I. Kumov (*Zh. Anal. Khim., SSSR*, 1954, **9** [4], 229–232).—Cadmium is pptd. in acid medium as a phenazone complex, $[C_{11}H_{12}ON_2]H_2[CdBr_4]$, $2C_{11}H_{12}ON_2$ (Gusev, *Zh. Anal. Khim., SSSR*, 1949, **4**, 175). To prepare the reagent, 5 g of phenazone and 10 g of KBr are dissolved in 85 ml of 0·25 *N* H_2SO_4. To determine Cd, an excess of the reagent is added and the mixture stirred to give a crystalline ppt., which is transferred to a filtering crucible, part of the filtrate being used to complete the transfer. The ppt. is washed once or twice with the diluted reagent solution (1 + 1) and immediately washed into a flask with 25 to 30 ml of water. After solution, 3 g of Na acetate are added and then 25 to 30 ml of 0·1 *N* iodine. The flask is stoppered, the contents are vigorously shaken, giving a dark brown ppt., which after 30 to 40 min. is dissolved by the addition of 25 to 50 ml of ethanol; the excess of iodine is titrated with thiosulphate. One ml of 0·1 *N* iodine is equivalent to 1·405 mg of Cd. The method is satisfactory in the presence of Mg, Al, Cu, Zn and Co, and can be used in the analysis of alloys. For Elektron, 1 g is dissolved in dil. (1 + 10) H_2SO_4 soln., and for bronze, 1 g is dissolved

in 10 ml of dil. (1 + 1) HNO_3 soln.; the solution is evaporated to dryness and 50 ml of water are added; for each the solution is then treated with the reagent as described above. Cadmium can also be determined by titration of the solution of the ppt. with 0·1 *N* NaOH, by using methyl orange as indicator. The error in both instances is within ± 3 per cent. of the content. G. S. SMITH

570. **Gravimetric determination of cadmium by means of salicylaldehyde thiosemicarbazone.** V. Hovorka and Z. Holzbecher (*Bull. Int. Acad; Tchéque, Sci. Cl. Math., Natur. et Méd.*, 1953, **51**, 43; *Referativnýĭ Zh., Khim.* 1954, Abstr. No. 20,357).—Cadmium is pptd. from soln. of the nitrate or sulphate by ethanolic salicylaldehyde thiosemicarbazone; a yellow crystalline salt, Cd $(C_8H_8ON_3S)_2$, containing 22·4 per cent. of Cd is formed. The method is not applicable to solutions containing Cl′, F′, or oxalate; pptn. is possible in the presence of Ca, Sr, Ba and Mg salts, but the results may be too high because of co-precipitation. E. HAYES

571. **Potentiometric determination of cadmium with diantipyrinylmethane.** V. P. Zhivopistsev (*Uch. Zap. Molotovskogo Gos. Un-ta*, 1953, **8** [1], 141–144; *Referativnýĭ Zh., Khim.*, 1954, Abstr. No. 22,152).—The method is based on the potentiometric determination of excess of Br′ after pptn. of $Cd^{\cdot\cdot}$ as $(C_{22}H_{24}O_2N_4)_2H_2[CdBr_4]$. *Procedure*—To a soln. containing 10 to 50 mg of Cd (free from halogens and significant amounts of HNO_3) in a 100-ml calibrated flask, add 2 to 3 ml of conc. H_2SO_4. Add 40 to 50 ml of 0·2 *N* KBr, heat to 80° to 90° C and add 0·4 to 0·6 g of diantipyrinylmethane, either solid or dissolved in 15 to 20 ml of 0·5 *N* H_2SO_4; make up to the mark with H_2O and set aside for 60 to 90 min. Make up to the mark again, mix well, filter the soln. and titrate the excess of bromide in an aliquot with 0·1 *N* $AgNO_3$, using a silver electrode. In the presence of Fe^{III} salts (< 8 to 10 per cent.), the pptn. of the complex is retarded and the soln. must be set aside for 2 to 3 hr. If large amounts of Fe^{III} are present, the acidified soln. is heated almost to boiling with sulphite and a few drops of diantipyrinylmethane soln.; the disappearance of the red colour indicates complete reduction of the Fe^{III}. One ml of 0·1 *N* KBr ≡ 0·00281 g of Cd. The relative error is less than 3 per cent. Copper, Zn, Fe, Co, Ni, Cr, Mn, Al, Mg and small amounts of alkaline-earth metals do not interfere. E. HAYES

572. **Detection of mercury by the reduction of alkali iodomercurates or cyanomercurates with formaldehyde.** A. Jílek and J. Brandštetr (*Chem. Listy*, 1954, **48** [3], 386–390).—The following procedure for the detection of Hg in the presence of the cations of the analytical groups I to V illustrates the use of this new and highly sensitive test. Treat the test soln., contained in a porcelain dish and acidified with HCl, with a few crystals of $KClO_3$ and heat it on a water-bath for 2 min. Disregarding any ppt., add 1 to 2 ml of a cold saturated soln. of tartaric acid, followed by an excess of 10 per cent. aq. KI and 20 per cent. aq. NaOH until the soln. is distinctly alkaline. Clarify the soln. with a few drops of a 5 per cent. soln. of KCN. If Ag is present, add a further excess of KCN. To the cool soln., add a few drops of 40 per cent. formaldehyde; the appearance of a dark grey ppt. of metallic Hg indicates the presence of Hg in the original soln. As little as 0·02 mg of Hg in 5 ml can readily be detected. Procedures for carrying out the test for Hg among cations of group I B, in the presence of $Mn^{\cdot\cdot}$, $Cr^{\cdot\cdot\cdot}$ and CrO_4'' and in cinnabar are also described. G. GLASER

573. **The use of hydrochloric acid solution of 8-hydroxyquinoline for the semimicro determination of aluminium.** S. M. Éfros and N. Z. Golýnko (*Tr. Leningr. Tekhnol. In-ta*, 1953, [27], 126–133; *Referativnýĭ Zh., Khim.*, 1954, Abstr. No. 25,757).—To 1 to 2 ml of soln. (4 to 8 mg of Al) are added 9 ml of the reagent (2 g of 8-hydroxyquinoline and 4 ml of HCl, sp. gr. 1·19, diluted with distilled H_2O to 100 ml). The solution is heated to 90° to 95° C, and ≃ 1·5 ml of 2 *N* Na acetate are added; heating is continued until the ppt. crystallises (≃ 5 min.). Excess of the reagent (≃ 3·5 ml) is added and, after 10 to 15 min., the ppt. is filtered, washed 3 or 4 times with hot water to remove Cl′, and dried to constant weight at 125° to 130° C. E. HAYES

574. **New fluorescence methods for the detection of aluminium.** S. Holzbecher (*Coll. Czech. Chem. Commun.*, 1954, **19** [2], 241–251).—The reactions of aluminium salts with salicylaldehyde and 2-hydroxy-1-naphthaldehyde and 18 of their derivatives are studied. Generally the reaction is accompanied by a marked increase in the intensity of fluorescence of the reagent in u.v. light; the aluminium salts of two of the reagents also fluoresce in visible light. The most suitable reagent for the detection of small quantities of Al is salicylidene-*o*-aminophenol, with which 0·005 μg of Al can be detected at a limiting concn. of 1 in 10^8. The test is most sensitive at pH 5; sensitivity decreases as the pH is lowered. If salts of Bi, Sb, Sn, Ti, Zr or Th are present, ammonium citrate must be added; the soln. is then neutralised to bromothymol blue by the addition of aq. NH_3 soln. and the pH is adjusted to 5 to 5·5 with acetic acid and sodium acetate. Salts of Ag, Pb, Tl^{I}, Cd, Mn, La^{III}, Ce^{III}, Ca, Sr, Ba, Mg, Na or K or ammonium salts have hardly any effect on the reaction, but the sensitivity is reduced by $UO_2^{\cdot\cdot}$ or salts of Cu, Bi, Hg^{II}, Sb^{III}, Sn^{II}, Zn, Cr, Ti^{IV}, Th, Zr, Ni, Co, Fe^{III} or Be. Chlorides, sulphates or borates do not interfere but fluorides quench the fluorescence. [This is a translation into Russian of a paper originally published in *Chem. Listy*, 1953, **47**, 680.] E. HAYES

575. **Quantitative determination of aluminium in silica gel activated with alumina.** K. G. Miesserov (*Zh. Prikl. Khim., SSSR*, 1954, **27** [6], 677–680).—Aluminium, present in silica gel as aluminosilicate only, is completely extracted with cold *N* HCl and determined by the usual methods. If free Al_2O_3 is present, the solid must be boiled with the acid; Treadwell's method of aluminium determination gives the best results. R. C. MURRAY

576. **Complete spectrographic analysis of aluminium alloys.** J. Kuba and M. Dvořák (*Hutnické Listy*, 1953, **8** [8], 405–408, 408a and 408b; *Referativnýĭ Zh., Khim.*, 1954, Abstr. No. 18,623).—A method is described for the simultaneous spectrographic determination of all the components of Al - Zn - Cu alloys, including Fe, Si, Pb, Mn and Mg. Excitation conditions necessary for reproducible results are given. E. HAYES

577. **The polarographic determination of gallium.** H. E. Zittel (*Dissert. Abstr.*, 1954, **14** [9], 1303).—Conditions for the accurate quant. polarographic determination of Ga have been examined and a routine procedure has been devised. Gelatin is

effective in suppressing the max. of the gallium wave. The value of i (diffusion current) is influenced greatly by pH, whilst a decrease in Ga concn. increases $E_{\frac{1}{4}}$ to a more positive value. A clearly defined gallium wave, free of interference from the following hydrogen wave, can be obtained by controlled addition of a suitable buffer and by limiting the Ga concn., i varying linearly with the latter. Only Cr ions interfere seriously. The dropping-mercury electrode can be used.

W. J. BAKER

578. **The separation of small quantities of gallium from iron by means of a strongly basic anion-exchange resin.** E. Blasius and M. Negwer (*Z. anal. Chem.*, 1954, **143** [4], 257–259).—The solution containing Ga and Fe is diluted to 150 ml, oxalic acid (1 g) is added and the solution is buffered to pH 4·0 with ammonium acetate. To the column (diam. 1·6 cm) of Permutit ES (30 ml) as the nitrate, washed with 0·001 *N* oxalic acid, add the solution at a rate of 4 to 5 ml per min. Wash the column with 0·001 *N* oxalic acid (50 ml) and reject all solutions which have passed through. Elute the Ga with *N* NaOH (300 ml) and estimate gravimetrically with hydroxyquinoline. Wash the column with water (50 ml) and elute the Fe with 2 *N* HNO_3 (300 ml) and estimate gravimetrically as the oxide.

P. S. STROSS

579. **Determination of carbon and sulphur in ferrous metals. A combined method.** A. C. Holler, R. Klinkenberg and C. Friedman (*Anal. Chem.*, 1954, **26** [10], 1658–1661).—A 1-g sample of steel or cast iron is mixed with tin powder (1 g of 30 mesh) and covered with a little alundum powder (90 mesh) in a small cupel. The sample is burnt in a stream of O (600 ml per min.) within a high-frequency furnace. The issuing gases and O used for flushing are passed in turn through a specially designed absorption-titration vessel containing H_2O_2, conc. H_2SO_4 and anhydrone tubes, a tube containing a catalyst, for conversion of CO to CO_2, and finally an absorbing tube containing soda-lime. The S is determined titrimetrically with standard NaOH (bromocresol green) or, alternatively, it is absorbed in water as SO_2 and determined iodimetrically, and the C gravimetrically, as CO_2, by standard procedures. Analytical results for 25 standard ferrous samples are given; average deviations of ± 0 to ± 0·002 per cent. for S and ± 0 to ± 0·01 per cent. for C are claimed.

D. A. PANTONY

580. **The absorptiometric determination of silicon in ferrous materials.** Glasgow Absorptiometric Panel (*Metallurgia*, 1954, **50**, 145–148).—An absorptiometric method suitable for the routine determination of Si over the range 0 to 4 per cent. in all types of plain carbon and alloy steels and cast irons is described. *Procedure*—Two 0·25-g samples of iron ore, one silicon-free, are dissolved in 100 ml of acid in 500-ml calibrated flasks and the volumes are made up to 500 ml. The acids used are (*i*) 3·5 per cent. v/v H_2SO_4 for plain carbon and low alloy steels, (*ii*) HCl - HNO_3 for all high alloy materials, and (*iii*) H_2SO_4 - HNO_3 for plain and alloy irons. When (*i*) or (*iii*) is used potassium permanganate and hydrogen peroxide are added, and the soln. is cooled, diluted to 500 ml and shaken thoroughly. With (*ii*), the permanganate is omitted and the solution is boiled. A 20-ml aliquot is taken from each soln., transferred into 50-ml calibrated flasks and treated as follows. To the test soln. are added 2·5 per cent. w/v ammonium molybdate soln. (10 ml) and, after standing, 5 per cent. oxalic acid soln. (10 ml); after mixing, 6 per cent. w/v ferrous ammonium sulphate soln. (5 ml) is added. The mixture is then diluted to 50 ml. and mixed thoroughly. To the compensating soln. are added oxalic acid soln. (10 ml), ammonium molybdate soln. (10 ml) and ferrous ammonium sulphate soln. (5 ml), mixing between each addition and finally diluting to 50 ml and mixing thoroughly. The optical density of each soln. is then measured on a Spekker absorptiometer with a mercury-vapour lamp, Ilford spectrum filters No. 606 and the most suitable size of cell, and compared with calibration graphs. A quicker and equally accurate procedure is outlined for the analysis of batches of samples having like Si contents.

G. C. JONES

581. **Rapid method of determining silica in Martin slag.** B. E. Reznik, G. P. Fedorova and G. N. Veretennikova (*Nauch. Zapisk. Dnepropetrovsk. Gos. Un-ta*, 1953, **43**, 79–84; *Referativnyĭ Zh., Khim.*, 1954, Abstr. No. 15,054).—The method is based on the photocolorimetric measurement of silico-molybdenum blue, obtained by the reduction of the yellow silico - molybdenum complex. The best reducing agent is ferrous ammonium sulphate. The colour of the complex is stable for over 10 min. Methods of dissolving the silica are examined and applied to different types of slag. For 9 to 25 per cent. of SiO_2, the error is 0·1 to 0·6 per cent.

E. HAYES

582. **Spectrophotometric determination of silica in steel and cast iron.** P. Enghag (*Jernkontorets Ann.*, 1954, **138** [7], 404–413).—A study is made of the effect of conditions on the method of Hill (*Brit. Abstr. C*, 1949, 375) and of Arnfeldt *et al.* (*Brit. Abstr. C*, 1952, 191). At room temp. 20 min. are required to complete the formation of molybdosilicic acid; at 100° C 2 min. are sufficient. As the compound formed rapidly changes to the more weakly coloured β form, it is important to adhere to the prescribed 1·5 ± 0·5 min. for heating, especially when the Si content is high. Reproducibility is better when the sample is dissolved by boiling with nitric acid, in preference to heating on a water-bath. Results obtained show fairly good agreement with gravimetric determinations.

G. MIDDLETON

583. **Photometric micro-determination of silicic acid.** J. Čelechovský (*Chem. Listy*, 1954, **48** [3], 391–396).—The proposed photometric method for the determination of SiO_2 is based on the conversion of SiO_2 to molybdosilicic acid and its reduction with $SnCl_2$ to molybdenum blue. *Procedure*—Treat the neutral solution, diluted to contain 0·001 to 0·28 mg of SiO_2 in 1 ml, with a 5 per cent. soln. of $(NH_4)_6Mo_7O_{24}.4H_2O$, add 0·05 per cent. aq. soln. of tropaeolin OO (1 to 2 drops) and acidify with 2 *N* HCl to a red colour. After 3 min., add 3 *M* citric acid (2 ml) to mask the excess of molybdate, followed, after a further 3 min., by 0·07 *N* $SnCl_2$ (0·12 ml). Dilute and measure the extinction at 750 mμ.

G. GLASER

584. **The colorimetric determination of titanium in iron and steel by use of sulphosalicylic acid.** K. Saarni and S. Suikkanen (*Z. anal. Chem.*, 1954, **143** [2], 112–113).—The method of Ziegler and Glemser (*Anal. Abstr.*, 1954, **1**, 54) is suitable for the determination of Ti in iron or steels if most of the Fe, Cr and Ni is first removed. This can be done by electrolysis with a mercury cathode, or by ether extraction.

P. S. STROSS

585. **Determination of germanium in the electrolytes used for zinc production.** E. Bertorelle, L. Canonica and A. Colombo (*Chim. e Ind.*, 1954, **36** [7], 534–535).—Ge is co-precipitated with Cu (added as $CuSO_4$) by sodium dithionate, mainly as sulphide; the ppt. is dissolved in dil. (1 + 3) nitric acid and chromatographed on paper, after eluting with acetone - ethyl methyl ketone - HCl ($d = 1{\cdot}18$) - water (85:5:5:5) and developing with a 0·05 per cent. soln. of phenylfluorone (2:3:7-trihydro-9-phenylfluorone) in 95 per cent. ethanol adjusted to pH 1 to 1·5 with 6 *N* HCl. Ge shows a characteristic red colour with an R_F value of 0·06. R. C. MURRAY

586. **Turbidimetric micro-determination of zirconium.** G. W. Leonard, jun., D. E. Sellers and L. E. Swim (*Anal. Chem.*, 1954, **26** [10], 1621–1623).—Five ml of a soln. containing 10 to 125 p.p.m. of Zr is made 0·25 *N* with respect to HCl soln., and mixed with 10 ml of phthalic acid reagent (7 g of potassium hydrogen phthalate in 1 litre of 0·25 *N* HCl). After digestion at 100° C for 10 min., the suspension is cooled and shaken and the turbidity is measured at 430 mμ. The Zr concn. is derived from a calibration curve prepared from standards. Standard deviation is given as ± 2 p.p.m. over the range of concn. examined. Interference from 18 ions is investigated; only PO_4''' and F' interfere seriously, and a procedure is suggested for the determination of F' concn. by measurement of the suppression of the zirconyl phthalate turbidity.

D. A. PANTONY

587. **Amperometric titration of thorium with fluoride.** M. Sundaresan and M. D. Karkhanavala (*Current Sci.*, 1954, **23** [8], 258–259).—An amperometric method is described for the determination of 0·6 to 10 mg of Th with an accuracy of ± 2 per cent. The Th solution (pH 2 to 2·4), freed from excess of mineral acids and their salts, is titrated at 0 volts *vs.* the S.C.E. with standard fluoride solution [prepared from pure NaF and standardised amperometrically by the method of Petrov and Nash (*Anal. Chem.*, 1950, **22**, 1274) or Langer (*Ind. Eng. Chem., Anal. Ed.*, 1940, **12**, 511)] in the presence of 0·2 *M* $NaClO_4$ as supporting electrolyte and 0·001 *M* $Fe(ClO_4)_3$ as the indicator (titre, corrected for added Fe, *V* ml). The capillary has a drop-time of 4·0 sec. and delivers 1·469 μg of Hg per sec. in a solution 0·2 *M* in $NaClO_4$, 50 per cent. in ethanol and 0·001 *M* in $Fe(ClO_4)_3$ at 0 volts *vs.* the S.C.E. ($m^{\frac{2}{3}}t^{\frac{1}{6}}$ equals 1·629 $mg^{\frac{2}{3}}$ $sec.^{\frac{1}{2}}$). The titration is performed in 50 per cent. ethanol. The end-point is reached when the current, which initially remains constant but decreases rapidly towards the end, finally becomes zero; in practice, titrant is added until the current becomes negative. The amount of Th is $58{\cdot}05 \times V \times M$ mg, where *M* is the molarity of the fluoride solution. S. C. JOLLY

588. **A photometric determination of tin with thiourea.** W. Nielsch and G. Böltz (*Z. anal. Chem.*, 1954, **143** [3], 161–167).—The extinction of the yellow complex formed between Sn and thiourea in HBr solution is independent of the concentration of HBr if this is between 14 and 16 per cent., but rises proportionately if the concentration of thiourea is > 6 per cent. The extinction increases with time, and measurements should be made between 20 and 30 min. after the addition of thiourea; small changes of temperature have little effect. From 5 to 80 μg of Sn per ml can be estimated.

P. S. STROSS

589. **Determination of tin in titanium alloys.** W. A. Dupraw (*Anal. Chem.*, 1954, **26** [10], 1642–1645).—A 1-g sample of titanium alloy containing 0·5 to 3 per cent. of Sn is dissolved by heating with water (60 ml), dil. (1 + 1) H_2SO_4 (10 ml) and fluoroboric acid (50 per cent.) (10 ml), and, if the presence of Mo is suspected, an excess of 30 per cent. aq. H_2O_2 is added when soln. is complete. The soln. is diluted with 100 ml of water, and Fe powder (5 g), 2 drops of 1 per cent. $SbCl_3$ in (1 + 5) HCl and conc. HCl (60 ml) are added. After heating until all the iron has dissolved, the soln. is boiled for 2 min. The resulting $Sn^{\cdot\cdot}$ is cooled under CO_2 and titrated with standard 0·01 or 0·1 *N* I' soln. or 0·02 *N* IO_3' soln. Results of analyses of 5 Ti alloys are given. No interference is caused by Mo, Ti and Cr, but V, W and large excesses of Mo give slightly high results; $Cu^{\cdot\cdot}$ and NO_3' interfere seriously. D. A. PANTONY

590. **Determination of tin, iron and molybdenum in titanium by paper chromatography.** I. Kolier and C. Ribaudo (*Anal. Chem.*, 1954, **26** [10], 1546–1549).—A titanium sample (0·5 g) is dissolved in ≃ 7 ml of hot conc. HCl. After cooling, 30 per cent. H_2O_2 is added until the soln. becomes yellow; 5 drops are then added in excess and the soln. becomes red. The mixture is digested on a steam-bath until the colour reverts to yellow, and the cooled soln. is made up to 10 ml. Five 0·1-ml aliquots are placed on separate 22 × 1·5-inch strips of Whatman No. 1 paper, and, after drying, the strips are treated in the standard downward-flow technique with *n*-butanol - HCl as developing solvent; a blank determination is run alongside. One of the air-dried chromatograms is sprayed with alizarin - ethanolic ammonia (0·5 per cent. alizarin in ethanol exposed to NH_3 vapour for 1 hr.) to locate the bands (R_F values are: Sn, ≃ 0·93; Fe, 0·50 to 0·60; Mo, 0·45 to 0·55). After comparison with this locating strip, appropriate sections are cut from the other strips for determination of Fe, Mo and Sn by standard methods. Sn is determined polarographically in H_2SO_4 - HCl - NH_4Cl soln. at −0·35 to −0·75 V; Fe, colorimetrically as $Fe^{\cdot\cdot}$ - 1:10-phenanthroline complex at 510 mμ, or polarographically in aq. NH_3 soln. - oxalic acid soln. at −0·05 to −0·45 V; Mo, colorimetrically as a complex with $Sn^{\cdot\cdot}$ and NH_4CNS in *iso*propyl ether at 475 mμ, or polarographically in NaOH - $HClO_4$ soln. at + 0·2 to −1·2 V. The choice of methods for Fe and Mo enables determination of these elements when their chromatographic bands overlap. Allowance is made for results obtained from the blank strip. Examination of the R_F values of 15 cations shows that they do not interfere.

D. A. PANTONY

591. **The anodic separation of lead as lead dioxide. (Interference by zinc and iron.)** F. Umland and K. Kirchner (*Z. anal. Chem.*, 1954, **143** [4], 259–264).—The optimal concentration of HNO_3 for the electrolytic separation of lead as the dioxide lies between 5 and 9 per cent. w/w, ≃ 2·5 V being applied. One g of urea for the lower acid concentration, and 2 g for the higher, should be added to destroy any HNO_2 formed. Zinc does not interfere generally, but amounts of Fe greater than half the quantity of Pb present interfere and must be removed before electrolysis. P. S. STROSS

592. **A rapid method for the determination of lead in steel.** G. H. Bush (*Analyst*, 1954, **79**, 697–702.)—A rapid method for the determination of Pb in

steel, alloy steel and cast iron is described. *Procedure*—The sample is dissolved in dil. HCl soln. in a flask fitted with a Contat - Göckel valve containing a sodium citrate and Na_2CO_3 soln. to exclude air during solution and provide an atmosphere of CO_2 after solution. The soln. is treated with KCN, brought to incipient boiling, treated with 1 drop of H_2O_2 to oxidise PbS, filtered and diluted to a definite vol. with cold water. The liquid is divided into two equal parts. One part (*a*) is treated with sodium dithionite and the other, (*b*), with Na_2S; after adjustment to definite vol., the absorption of each is measured with the Spekker absorptiometer with an Ilford 601 violet filter against a water setting at 1·0. The lead content is ascertained from the difference between the absorptions of (*a*) and (*b*) by proportionality from similar figures determined with a standard steel of known lead content. Alternatively the absorption of soln. (*b*) may be measured against that of (*a*) in a spectrophotometer at 430 mμ. The value for the optical density varies as the amount of Pb present, which is evaluated by reference to similar figures for standard steel of known lead content.

A. O. JONES

593. **A method for the volumetric determination of ammonia with 0·1 *N* potassium bromate solution. [Preliminary communication].** D. Köszegi and É. Salgó (*Z. anal. Chem.*, 1954, **143** [6], 423–424).—A modification of the procedure studied by Kolthoff and Laur (*Brit. Abstr. A*, 1928, 498) for the titration of ammonia and ammonium salts with hypobromite is briefly described. Quantitative results are claimed. Standard potassium bromate solution (in excess) is used as a source of bromine, which is converted into hypobromite immediately before reaction with the sample; the excess is titrated iodimetrically with thiosulphate. A. R. ROGERS

594. **Chromatographic semi-micro analysis of gases. IX. Determination of nitrous oxide.** J. Janák and M. Rusek (*Chem. Listy*, 1954, **48** [3], 397–400).—The chromatographic technique of gas analysis is suitable for the rapid estimation of N_2O in admixture with hydrogen, nitrogen, methane and ethane, in commercial N_2O and in natural gas. The R_F values of N_2O, measured in the usual way in an adsorption column 220 mm long and 5·1 mm in diameter with the eluting gas (CO_2) passing at the rate of 0·7 ml per sec., were as follows: on activated carbon, 0·0264 at 20° C and 0·234 at 80° C; and on silica gel, 0·0528 at 20° C and 0·297 at 80° C. Owing to the slight solubility of N_2O in conc. KOH, the nitrometer should be filled with a KOH soln. that has been exposed to an atmosphere containing N_2O. G. GLASER

595. **Analysis for industry. [Determination of phosphates.]** D. Haywood (*Ind. Chem.*, 1954, **30**, 499–500).—Recent methods for analysing condensed inorganic phosphates, particularly $Na_5P_3O_{10}$ containing $Na_4P_2O_7$, $(NaPO_3)_3$, $(NaPO_3)_6$, Na_2HPO_4, NaCl, Na_2SO_4 and Na_2CO_3 are described. In one method, $Na_5P_3O_{10}$ and $Na_4P_2O_7$ are separated as Zn salts, which are then analysed for Zn and H_2O content, from which pyrophosphate and triphosphate are calculated. The remaining salts are determined by known methods. Ortho-, meta-, pyro- and triphosphate can also be separated by adding $BaCl_2$ to a solution at pH 0·5, removing $BaSO_4$ and then selectively pptg. $Ba_3P_6O_{18}$ at pH 2·5, $Ba_5(P_3O_{10})_2$ at pH 3·4, $Ba_2P_2O_7$ at pH 4·0, and $BaHPO_4$ at pH 8·0, leaving $Na_3P_3O_9$ in solution. The ppt. are then hydrolysed to orthophosphate and determined in a known manner. The application of an isotope-dilution technique is also described. D. R. PECK

596. **The precipitation of trimetaphosphate and tetrametaphosphate anions with benzidine hydrochloride.** H. Hecht (*Z. anal. Chem.*, 1954, **143** [2], 93–102).—Trimetaphosphate is separated from other phosphates by precipitation with benzidine hydrochloride and determined colorimetrically after hydrolysis to phosphate. Results are 5 to 6 per cent. low owing to the appreciable solubility of the benzidine - metaphosphate complex. The latter has no constant composition. Barium ions in low concn. do not interfere. *Procedure*—To an approx. 0·002 *M* solution of trimetaphosphate, $Na_3P_3O_9$, add benzidine solution (7 ml) [benzidine (20 g) and conc. HCl (30 ml), made up to 500 ml with water] and adjust the pH to 5·0 with NaOH solution. Remove the precipitate by filtration on sintered glass, wash it with cold water and dry it to constant weight at 95° C. Dissolve an accurately weighed portion of the dry precipitate in hot 5 per cent. HCl (60 ml), cool to 0° C, and precipitate the benzidine with 10 per cent. H_2SO_4 (50 ml). Filter, precipitate the excess of sulphate ions in the filtrate with barium nitrate solution and hydrolyse the trimetaphosphate to phosphate for 2 hr. under reflux with conc. HCl (10 ml). Determine the phosphate colorimetrically. The method also works well for tetrametaphosphate. P. S. STROSS

597. **The separation of phosphoric acid ions from cations of the analytical groups I, II and III with the aid of ion-exchange resins.** I. G. Lakomkin (*Tr. Leningr. Tekhnol. In-ta*, 1953, [27], 3–28; *Referativnyĭ Zh., Khim.*, 1954, Abstr. No. 25,730).—Phosphoric acid ions are separated from a filtered soln. (pH < 2) by passage through a column of Wofatit KS or Ionite SBS (hydrogen forms) and washing with water. The cations are then displaced by washing with HCl soln. The method permits the separation of a fraction of a milligram of the cations of groups II and III in the presence of 5000 times as much PO_4'''. E. HAYES

598. **The iodimetric determination of arsenic in ten-milligram amounts of organic compounds.** G. Bähr, H. Bieling and K.-H. Thiele (*Z. anal. Chem.*, 1954, **143** [2], 103–111).—The method described is suitable for routine analysis of all organic arsenic compounds, including those that give low results by the classical wet oxidation methods with sulphuric and nitric acids (Wintersteiner, *Mikrochemie*, 1926, **4**, 155); accuracy is 0·1 per cent. *Procedure*—Weigh 10 mg of sample, or sufficient to give a titre of 11 ml of 0·01 *N* thiosulphate, into a Kjeldahl flask, add conc. H_2SO_4 soln. (1 ml) and set aside for 5 min.; dissolve the sample if necessary by warming, cool and add small quantities of $KMnO_4$, warming gently after each addition until the dirty green of the solution disappears and the precipitated MnO_2 no longer dissolves. Add water (2 ml) and 30 per cent. H_2O_2 (2 to 3 drops) and heat until a clear solution is obtained. (if a clear solution is not obtained, heat until fumes of SO_3 appear and repeat oxidation with $KMnO_4$; this is always necessary with cacodylic acid.) Heat until fumes of SO_3 appear, cool, add water (1 ml) and heat until fumes of SO_3 again appear, adding water (1 ml); repeat this procedure thrice more. Add water (1 ml) and wash the sample into a flask with oxygen-free and chlorine-free conc. HCl soln. (2 × 2·5 ml),

add freshly prepared 4 per cent. KI solution (2 ml), shake, set aside for 20 min. in the dark and titrate with 0·01 *N* thiosulphate solution. Subtract the reading obtained from that of a blank similarly treated. The method has been tested on a wide range of compounds and found to be satisfactory. Results were high with phenylarsonic acid, because it loses one molecule of water when dried at 120° to 130° C. P. S. Stross

599. **Iodimetric determination of arsenic acid by use of organic solvents.** G. B. Shakhtakhtinskii (*Zh. Anal. Khim., SSSR*, 1954, **9** [4], 233–238).—The method is based on the displacement of the equilibrium of the reaction $H_3AsO_4 + 2I' + 2H \leftrightarrows H_3AsO_3 + H_2O + I_2$ from left to right, by extraction of the I with an organic solvent. *Procedure*—To 10 ml of As^V solution add 10 to 12 ml of dil. H_2SO_4 soln. (1 + 1), 20 ml of chloroform or benzene, and 3 ml of *N* KI. For As concn. > 0·1 *N*, increase the amount of KI. Shake the mixture for a few seconds, dilute with an equal vol. of water and titrate the iodine with thiosulphate.

G. S. Smith

600. **Modified method for the volumetric determination of arsenic and antimony in mixtures, white metal (anti-friction alloy) and antimony sulphide ores.** A. Jílek and M. Nedorost (*Chem. Zvesti*, 1953, **7** [1–2], 19–31; *Referativnýĭ Zh., Khim.*, 1954, Abstr. No. 18,592).—A modification of the method of Evans (*Analyst*, 1929, **54**, 523) is described. The mixed salts of As, Sb^{III} and Sn^{IV} in HCl soln. are treated with NaH_2PO_2. The ppt. of reduced As is titrated with 0·1 *N* $KBrO_3$. For white metal or antimony sulphide ores (containing As, Sb, Cu, S, Fe_2O_3, Al_2O_3, SiO_2), the sample is first decomposed with conc. H_2SO_4 and the As in the filtrate is reduced as above. The method is applicable to the determination of As in the presence of Se and Te in sludge from sulphuric acid works. Two methods are described for determining Sb in the presence of As and Sn, and in alloys and ores. In the first method, As is removed by reducing with NaH_2PO_2, and Sb is pptd. from the filtrate with H_2S, together with the sulphides of other metals present; the sulphides are then dissolved in conc. H_2SO_4 and the Sb^{III} is titrated with 0·1 *N* $KBrO_3$. In the second method, the As is determined as described in an aliquot of the soln.; in a second aliquot all the sulphides are pptd. with H_2S and dissolved in conc. H_2SO_4; total As^{III} plus Sb^{III} is titrated with 0·1 *N* $KBrO_3$, and the amount of Sb is found by difference.

E. Hayes

601. **The photometric determination of antimony by means of thiourea.** W. Nielsch and G. Böltz (*Z. anal. Chem.*, 1954, **143** [2], 81–93).—The photometric determination of Sb with thiourea is studied in HCl and HBr solutions; the extinction maximum lies below 320 mμ, but, although the yellow colour is unsuitable for measurement on a filter instrument, it can be measured with sufficient accuracy by using the mercury line at 366 mμ, if the temperature is controlled. Hydrogen bromide solutions are more suitable than HCl, and the optimum concn. is 10 per cent. Ten per cent. or more of thiourea is advisable. Beer's Law is obeyed for 1 to 25 μg of Sb per ml. Bi and Sn interfere. P. S. Stross

602. **Extraction and photometric determination of antimony by means of rhodamine B.** W. Nielsch and G. Böltz (*Z. anal. Chem.*, 1954, **143** [4], 264–272).—The antimony - rhodamine B complex is selectively extracted from aqueous acid solution containing an excess of rhodamine B with benzene or di*iso*propyl ether, preferably the latter. The extinction of the solvent phase (maximum 550 to 560 mμ) is independent of temp., but varies with HCl concn. of the aqueous phase; extraction is complete after a single 30-second shake-out. The Beer - Lambert law is obeyed for Sb concn. of 1 to 20 μg of Sb per ml of solvent. P. S. Stross

603. **Studies on the determination of bismuth with 8-hydroxyquinoline.** A. Jílek and M. Křivánek (*Chem. Zvesti*, 1953, **7** [9], 563–569; *Referativnýĭ Zh., Khim.*, 1954, Abstr. No. 23,970).—Bismuth is pptd. from a buffer soln. containing acetic acid - Na acetate or ammonium tartrate. With the latter, Bi can be determined in the presence of alkali metals, As, W, Te or Se; chloride ions do not interfere. The bismuth - hydroxyquinoline complex is dried at 105° C and weighed as $Bi(C_9H_6ON)_3.H_2O$. At pH 5·15 to 10·55, the pptn. is quantitative.

E. Hayes

604. **Photocolorimetric determination of bismuth in lead with thiourea.** N. N. Aleshkina (*Sb. Stud. Nauchn. Rabot Rostovskogo Gos. Un-ta*, 1953, [2], 92–95; *Referativnýĭ Zh., Khim.*, 1954, Abstr. No. 16,801).—The colour of the complex formed by the action of 1 per cent. thiourea soln. on Bi obeys Beer's law for 1 to 6 mg of Bi per litre. The concn. of HNO_3 in the soln. should be 0·5 to 1 *N*, and the concn. of lead should be less than 1 per cent. In the separation of Bi from Pb, the best results are obtained by pptn. of the Bi with Na_2HPO_4, followed by solution of the ppt. in HNO_3. E. Hayes

605. **The photometric determination of bismuth with thiourea. III. In HBr solution.** W. Nielsch and G. Böltz (*Z. anal. Chem.*, 1954, **143** [3], 168–172).—It is convenient to carry out determinations in HBr solutions, as most Bi alloys are soluble in HBr. The extinction of the yellow complex between Bi and thiourea is independent of time and small changes in temperature, but is dependent on the concentrations of HBr and thiourea. For a suitable filter, it is possible to use reagent strengths such that small changes in their concentrations have no effect on the extinction. From 3 to 20 μg of Bi can be estimated. P. S. Stross

606. **Determination of small amounts of vanadium in aluminium and clay.** L. Erdey, K. M. Vigh and L. Mázor (*Acta Chim. Hung.*, 1954, **4** [2–4], 259–270).—The Hungarian standard method for the determination of V in smelter Al and Al alloys, which consists in preparing, in an acid solution a yellow-coloured complex of vanadium molybdophosphate and measuring its extinction value, is improved in sensitivity by ≃ 12 per cent., by altering the composition of the acid mixture to H_3PO_4 (*d* 1·7, 300 ml) and H_2SO_4 (*d* 1·8, 100 ml) per litre. The improved method is suitable for the determination of V in smelter Al, but not for V in clay. The diphenylamine method, the 8-hydroxyquinoline-sulphonic acid process and the 8-hydroxyquinoline method cannot be used for the determination of V in smelter Al or in clay. Enrichment by Na diethylthiocarbamate is an advantage for the determination of V in clay. The HCl solution of processed alumina is adjusted to ≃ 3·0 pH; a V - diethylthiocarbamate complex is prepared in this solution and extracted by chloroform. The complex is decomposed by a mixture of HNO_3 and H_2O_2, and the V salt liberated is transferred to an aq. phase. The solution of V salt

obtained by the enriching process is evaporated to dryness. Thus the excess of acid is removed, and it may be possible to dissolve the V salt in a vol. appropriate to the quantity of V present. Colorimetric determination of V is then effected as vanadium molybdophosphate or tungstophosphate or, if Ti is absent, as a peroxo - vanadium complex. H. WREN

607. **The determination of sulphur in pyrites by the method of Grote and Krekeler.** H. Wiele (*Z. anal. Chem.*, 1954, **143** [4], 275–279).—Differences between results obtained gravimetrically and volumetrically on the same solution by the method of Grote and Krekeler (*Angew. Chem.*, 1933, **46**, 106) are due to occlusion of Na_2SO_4 on precipitated $BaSO_4$. The difficulty is overcome by using triethanolamine instead of NaOH for the pptn. The H_2O_2 solution need not be alkaline; the H_2SO_4 formed from the SO_2 should be titrated with triethanolamine by use of methyl red indicator. A subsequent gravimetric determination of SO_4'' gives correct results. P. S. STROSS

608. **The use of cation-exchange resins for the determination of sulphur in pyrites.** V. B. Aleskovskiĭ and Z. I. Kheifets (*Tr. Leningr. Tekhnol. In-ta*, 1953, [27], 121–125; *Referativnyĭ Zh., Khim.*, 1954, Abstr. No. 23,991).—Pyrites is decomposed by HCl plus HNO_3 and Fe''' is removed by the acid form of the cation-exchange resin SBS; the SO_4'' is then determined by pptn. with $BaCl_2$. The results are 0·4 to 0·7 per cent. higher than those obtained by the method in which the Fe^{III} is reduced by aluminium powder. E. HAYES

609. **Direct titrimetric determination of sulphate.** J. S. Fritz and M. Q. Freeland (*Anal. Chem.*, 1954, **26** [10], 1593–1595).—The sample, containing 2 to 4 millimoles of SO_4'', is dissolved in water (45 ml); to the soln. is added methanol (40 ml), and the pH is adjusted to 3 to 3·5 with 0·25 *M* Mg acetate or $HClO_4$. Standard Ba'' soln. (0·1 *M* $BaCl_2$ or $Ba(ClO_4)_2$ at pH 3 to 3·5 standardised against 0·01 *M* H_2SO_4) is run in until $\simeq$ 90 per cent. equivalence, when 5 drops of aq. Alizarin red S soln. (0·2 per cent.) or Thorin (0·025 per cent.) are added as indicator. The titration is completed slowly, the end-point being marked by the first permanent pink colour. Interfering ions can be removed by passage of the soln. through a cation-exchange column, Dowex 50 (H) prepared with 3·5 *N* HCl. The procedure is amenable to scaling down to the 0·2-millimole level. Effects of solvent, pH, speed of titration and temp. are investigated, and the interference due to NO_3', Cl', Br' and ClO_4' is discussed. In the absence of interfering ions, the precision is claimed to be as high as that obtained in the gravimetric $BaSO_4$ method, and there is a considerable saving in time. D. A. PANTONY

610. **Volumetric determination of sulphate and analysis of pyrites. Application of cation-exchange resins.** R. A. Whiteker and E. H. Swift (*Anal. Chem.*, 1954, **26** [10], 1602–1605).—After examination of the conditions which influence the titrimetric determination of SO_4'', the following procedure for determination of S in pyrites is evolved. The S in a 1-g sample is oxidised with a mixture of HNO_3 (30 ml), HCl (9 ml) and Br (15 drops), and the soln. is evaporated nearly to dryness and then evaporated twice more with conc. HCl (5 ml and 2 ml). The residue is dissolved in water (100 ml) and the soln. is filtered, the filtrate being made up to 250 ml with water. In a 25-ml aliquot, the H^+ concn. is determined by titration with 0·2 *N* NaOH to pH 6·5 to 7·5 (pH meter), and the halogen concn. by titration with 0·1 *N* $AgNO_3$ (dichlorofluorescein); the SO_4'' concn. is found by difference. Cations are removed before titration by passage of the 25-ml aliquot, diluted once with water, through a resin column (Amberlite IR–120, 18 to 40 mesh) that has been treated with 6 *N* HCl and then washed with water until the pH of the eluate is > 4. Results are compared with those from a standard gravimetric procedure: they are 0·2 per cent. lower and have a standard deviation of $\pm$ 0·12 per cent. A 50 per cent. saving in time is claimed. D. A. PANTONY

611. **Microchemical detection of sulphite, thiosulphate, sulphide, hydrogen peroxide and formaldehyde.** V. Hovorka and Z. Holzbecher (*Bull. Int. Acad. Tchéque Sci. Cl. Sci. Math., Natur. et Méd.*, 1953, **51**, 127; *Referativnyĭ Zh., Khim.*, 1954, Abstr. No. 22,178).—The method is based on the reduction of MnO_2 to Mn''. A drop of the test soln. on paper is covered with a thin layer of MnO_2, which is then dipped in benzidine acetate. If the soln. contains SO_3'', S_2O_3'', S'' or H_2O_2, a white spot is formed where the drop of soln. has been placed and the remainder of the paper turns blue. Formaldehyde gives a bright green spot on a blue background. In the presence of Cl', Br', NO_3', SO_4'', F', SiO_3'', MoO_4'', WO_4'', tartrate, citrate and oxalate, the sensitivity of the test is partially reduced, these ions producing violet spots. E. HAYES

612. **Peroxy compounds. I. Iodimetric determination of persulphuric acid in the presence of hydrogen peroxide. (Chlorine method.)** E. Schulek, L. Pungor and J. Trompler (*Acta Chim. Hung.*, 1954, **4** [2–4], 393–404).—A measured vol. of solution ($\equiv$ 0·7 to 1·4 mg of H_2O_2 or 5 to 10 mg of $H_2S_2O_8$) is diluted to $\simeq$ 20 to 25 ml with water, until the pH exceeds 1, in a glass-stoppered 100-ml Erlenmeyer flask, and then shaken with 3 ml of freshly saturated bromine-free chlorine water. After 30 sec., freshly prepared 5 per cent. aq. KCN (1 ml) is added and the mixture is shaken for 3 min. with frequent release of the stopper. After the addition of 10 per cent. H_2SO_4 ($\simeq$ 1 ml), CO_2 (washed by 5 per cent. $KHCO_3$ and 0·1 *N* $KMnO_4$) is passed through the solution for 20 min. to remove air and the bulk of the cyanogen chloride. Potassium iodide (0·5 g) and $(NH_4)_2SO_4$ (4 g) are added and the loosely stoppered flask is heated at 50° C for 5 min.; the liberated I is titrated in the cooled solution with 0·01 *N* $Na_2S_2O_3$ and starch as indicator. If H_2O_2 and $H_2S_2O_8$ are to be determined in the same solution, the sample (measured as described above) is diluted to $\simeq$ 20 to 25 ml, treated with 90 per cent. H_2SO_4 (0·75 ml), followed by KI (0·5 g), $(NH_4)_2SO_4$ (4 g), and 1 drop of 5 per cent. ammonium molybdate. The solution is heated for 5 min. at $\simeq$ 50° C in the loosely stoppered flask, cooled, diluted to $\simeq$ 80 ml and titrated with 0·01 *N* $Na_2S_2O_3$, with starch as indicator. H. WREN

613. **Peroxy compounds. II. Iodimetric determination of persulphuric acid in the presence of hydrogen peroxide. (Bromine method.)** E. Schulek, J. Trompler and E. Pungor (*Acta Chim. Hung.*, 1954, **4** [2–4], 405–410).—The sample (0·7 to 1·4 mg of H_2O_2 or 5 to 10 mg of $H_2S_2O_8$) is diluted to $\simeq$ 20 to 25 ml with water in a glass-stoppered 100-ml Erlenmeyer flask, and shaken with bromine water; excess of Br is removed by adding 1 per cent. hydrazine dropwise. One or two drops of methyl

orange are added to the colourless solution, which is then treated gradually with ≃ 0·01 *N* bromine water until the red colour of the indicator disappears. After addition of 1 to 2 drops of 5 per cent. phenol, the mixture is thoroughly shaken. Alternatively, most of the Br can be removed with hydrazine and the remainder immediately with phenol. After 5 min., KI (0·5 g) and $(NH_4)_2SO_4$ (4 g) are added, the solution is acidified with 10 per cent. H_2SO_4 (≃ 0·75 ml), heated at ≃ 50° C for 5 min., cooled, and the separated I is titrated with 0·01 *N* $Na_2S_2O_3$ in the presence of starch. The method is valid for 0·1 *N* solutions. The simultaneous determination of H_2O_2 and $H_2S_2O_8$ is carried out as described in Abstract 612 above. H. WREN

614. **Peroxy compounds. III. Iodimetric determination of persulphuric acid in the presence of hydrogen peroxide. (Thiocyanate method.)** E. Pungor, E. Schulek and J. Trompler (*Acta Chim. Hung.*, 1954, **4** [2–4], 411–416).—The sample (≡ 0·7 to 1·4 mg of H_2O or 5 to 10 mg of $H_2S_2O_8$) is diluted to ≃ 20 ml with water in a glass-stoppered 100-ml Erlenmeyer flask and treated with 10 per cent. H_2SO_4 (0·2 ml), 0·1 *M* KCNS (≃ 4 ml) and 1 drop of 5 per cent. ammonium molybdate. The liquid is shaken and kept for 3 min. with frequent loosening of the stopper. It is made alkaline for a few seconds with 4 per cent. NaOH (1 ml) and then acidified with 10 per cent. H_2SO_4 (≃ 1 ml). After addition of KI (≃ 0·5 g) and $(NH_4)_2SO_4$ (4 g), the loosely stoppered flask is heated at 50° C for 5 min., then cooled, and the liberated I is titrated with 0·01 *N* $Na_2S_2O_3$ in the presence of starch solution (which is added towards the end of the titration after dilution with ≃ 50 ml of water). The sequence of addition of reagents is important. The use of 0·1 *N* solutions is not recommended. H. WREN

615. **Peroxy compounds. IV. Iodimetric determination of permonosulphuric (Caro's) acid and hydrogen peroxide in the presence of one another. (Thiocyanate method.)** E. Pungor, E. Schulek and J. Trompler (*Acta Chim. Hung.*, 1954, **4** [2–4], 417–422).—The sample (≡ 0·7 to 1·4 mg of H_2O_2 or 0·6 to 6 mg of H_2SO_5) is diluted to ≃ 20 to 25 ml of water in a glass-stoppered 100-ml Erlenmeyer flask, acidified with 10 per cent. H_2SO_4 (≃ 2 ml) and treated with 0·1 *N* KCNS (≃ 0·5 ml). After ≃ 15 sec., KI (≃ 0·2 g) is dissolved in the solution, which is well shaken, and then treated with 1 drop of 5 per cent. ammonium molybdate soln. The liberated I is titrated with 0·01 *N* $Na_2S_2O_3$ in the presence of starch solution (added towards the end of the titration, after dilution with ≃ 50 ml of water). Another sample is similarly acidified, then treated with KI (≃ 0·5 g) and 1 drop of molybdate solution. The I, liberated by H_2O_2 and H_2SO_5, is titrated with 0·01 *N* $Na_2S_2O_3$ in the presence of starch. The method can only be used with 0·01 *N* solutions. H. WREN

616. **Peroxy compounds. V. Iodimetric determination of permonosulphuric and persulphuric acid and of hydrogen peroxide in the presence of one another. (Thiocyanate method.)** E. Pungor, E. Schulek and J. Trompler (*Acta Chim. Hung.*, 1954, **4** [2–4], 423–428).—For the determination of total H_2SO_5, $H_2S_2O_8$ and H_2O_2, the sample (≡ 1·2 mg of O) is diluted with water to ≃ 20 to 25 ml in a glass-stoppered 100-ml Erlenmeyer flask and acidified with 10 per cent. H_2SO_4 (≃ 1 ml). After addition of KI (0·5 g), $(NH_4)_2SO_4$ (4 g) and one drop of 5 per cent. ammonium molybdate solution, the solution is warmed at ≃ 50° C for 5 min., and the liberated I is titrated in the cooled solution with 0·01 *N* $Na_2S_2O_3$ in the presence of starch solution. For the determination of $H_2S_2O_8$ plus H_2O_2, the sample, measured and diluted as described above, is shaken after the addition of 10 per cent. H_2SO_4 (≃ 0·2 ml) and 0·1 *M* KCNS (≃ 0·5 ml). After ≃ 15 sec., KI (0·5 g) is added and the solution is well shaken, treated with 1 drop of 5 per cent. ammonium molybdate solution followed by $(NH_4)_2SO_4$ (4 g), warmed at 50° C and cooled. The liberated I is titrated with 0·01 *N* $Na_2S_2O_3$ in the presence of starch solution (added towards the end of the titration and after dilution with ≃ 50 ml of water). For the determination of $H_2S_2O_8$, the sample, measured and diluted as described above, is treated with one drop of 5 per cent. ammonium molybdate solution and 0·1 *M* KCNS (≃ 4 ml). After 3 min., the solution is made alkaline for a few sec. with 4 per cent. NaOH (1·5 ml) and re-acidified with 10 per cent. H_2SO_4 (≃ 1 ml). Potassium iodide (0·5 g) and $(NH_4)_2SO_4$ (4 g) are dissolved in the mixture, which is heated at ≃ 50° C for ≃ 5 min. The liberated I is titrated in the cooled solution with 0·01 *N* $Na_2S_2O_3$ in the presence of starch solution. The method is only recommended for use with 0·01 *N* solutions. The sequence of addition of the reagents is important. The composition of the anode liquid in H_2O_2 manufacture is thus determined; after 24 hr. the solution contains almost pure H_2SO_5. H. WREN

617. **Colorimetric determination of quadrivalent tellurium in the presence of sexavalent tellurium, and the determination of total tellurium.** A. Jilek and J. Vřeštál (*Chem. Zvesti*, 1953, **7** [1–2], 33–39; *Referativnyĭ Zh., Khim.*, 1954, Abstr. No. 18,610).—The method is based on the colorimetry of the yellow complex formed by $Te^{\cdots\cdot}$ with thiourea in acid soln. *Determination of* $Te^{\cdots\cdot}$ *in the presence of* $Te^{\cdots\cdots}$—To the test soln., add 5 ml of 4 *N* $HClO_4$ and 2 ml of 1 per cent. thiourea soln. and make up to 20 ml with water. Measure the colour immediately in a colorimeter fitted with a dark-blue filter. The method is applicable when $Te^{\cdots\cdots}$ is present in 100-fold excess. When the $Te^{\cdots\cdots}$ content is small (equal to or less than the $Te^{\cdots\cdot}$), 8 per cent. thiourea soln. and 0·8 *N* $HClO_4$ are used. *Determination of total tellurium*—To a soln. of the sample in a 25-ml calibrated flask, add 10 ml of 20 per cent. thiourea soln. in 2 *N* $HClO_4$; dilute almost to the mark with water and heat to boiling for 10 min. Cool to room temp. and make up to 25 ml; filter from the ppt. formed and measure the colour of the filtrate. Calculate the Te content with the aid of a calibration curve. The rate of reduction of $Te^{\cdots\cdots}$ increases with the thiourea concn. and with increase in temp. The mean error in determining total Te (3 to 9 mg of sample) is 1 per cent. E. HAYES

618. **Iodimetric determination of tervalent chromium.** E. Schulek and M. Szakács (*Acta Chim. Hung.*, 1954, **4** [2–4], 457–460).—The sample (≡ 0·2 to 3·5 mg of Cr) is diluted with water to ≃ 20 ml in a glass-stoppered 100-ml Erlenmeyer flask, made alkaline with 10 per cent. NaOH (1 to 2 ml), treated with 3 per cent. H_2O_2 (2 to 6 drops) and heated to boiling. Freshly saturated chlorine water (≃ 3 to 7 ml) is added to the cooled solution, which is vigorously shaken. After 3 min., the mixture is treated with freshly prepared 5 per cent. KCN (1 to 2 ml) and shaken, with frequent release of the glass stopper. After 2 min., 50 per cent.

H_2SO_4 (10 ml) is poured into the solution, which has been diluted to 40 ml, $KHCO_3$ (2 g, preferably as large crystals) is added and the flask is loosely stoppered. When gas evolution ceases, KI ($\simeq$ 0·2 g) is introduced, and, after 5 min., the liberated I is titrated with 0·01 *N* $Na_2S_2O_3$ in the presence of starch. H. WREN

619. **The use of potassium fluoride for the gravimetric determination of chromium.** Sh. T. Talipov and T. I. Fedorova (*Tr. Sredneaziatskogo Gos. Unta, Khim. Nauk*, 1953, No. 40, Kn. 5, 57–63; *Referativnyĭ Zh., Khim.*, 1954, Abstr. No. 23,972).—Chromium is determined gravimetrically as $K_2CrF_5.H_2O$. *Procedure*—The soln. under examination is heated to 70° to 80° C and a hot soln. of KF is added; after stirring, the soln. is set aside for 15 to 20 min. The ppt. is filtered off, then washed with 2 per cent. KF soln., ethanol and ether, and finally dried in air to constant weight. Small quantities of Ni or Co do not interfere. The method is equal in precision to existing methods for the determination of Cr. E. HAYES

620. **Micro-analysis by means of ion-exchange resins. II. Detection of small amounts of chromium with hydrogen peroxide.** M. Fujimoto (*Bull. Chem. Soc. Japan*, 1954, **27** [6], 347–350).—Chromium may be detected from the colour produced in resin grains by the product of the reaction between dichromate and H_2O_2. *Procedure*—Dichromate soln. (0·02 ml) is transferred by a capillary pipette to a spot-test plate and a few grains of 30-mesh Amberlite IRA–411 (Cl) resin are added. After 3 to 5 min. a drop of *N* H_2SO_4 soln. is added, followed by 1 per cent. H_2O_2 soln. The resin grains turn from bright yellow to grey, violet blue or violet brown, and the colour is observed as previously reported (*Anal. Abstr.*, 1954, **1**, 2098). The method allows 0·3 μg of Cr to be detected at a concn. of 1 in 10^5. The method is valid in the presence of a large excess of Fe^{++}, Fe^{+++}, Ni^{++}, Co^{++}, Cu^{++}, Mn^{++}, UO_2^{++} and acetic acid. If Cr is present as Cr^{+++}, oxidation is best carried out by heating the sample with a drop of a saturated soln. of $KMnO_4$, the excess of reagent being destroyed with H_2O_2. If 6 *N* acetic acid is used instead of *N* H_2SO_4, the clarity of the test is improved, but the sensitivity remains the same. J. H. WATON

621. **Rapid photometric method for the determination of small amounts of chromium in metallic aluminium.** L. Erdey and J. Inezédy (*Acta Chim. Hung.*, 1954, **4** [2–4], 289–301).—Metallic Al (1 g), water (50 pt.), conc. H_2SO_4 (3 pt.), conc. H_3PO_4 (1 pt.) and 0·5 per cent. $HgSO_4$ (3 ml) are placed in a 200-ml Erlenmeyer flask covered with a small funnel. When the reaction slackens, the flask is heated on a water-bath until solution is complete. The solution is evaporated to a syrup which is warmed with water until a clear solution is obtained; a slow stream of H_2S is passed through the clear solution for $\simeq$ 10 min. The solution is kept covered for some time, then boiled, cooled, made up to 100 ml with water and filtered through a dry hardened paper. Fifty ml of the filtrate are boiled for 10 to 15 min. after the addition of 1 per cent. $AgNO_3$ (1 ml) and 5 per cent. $(NH_4)_2S_2O_8$ (4 ml), and the hot solution is treated with a few crystals of $NaNO_3$ to destroy any $HMnO_4$ that may have formed. The cooled solution is washed, with as little water as possible, into a 50-ml flask, treated with freshly prepared 0·25 per cent. diphenylcarbazide in aq. acetone (1 + 1) and measured within 2 to 3 min. in a Pulfrich photometer using an S53 filter. Distilled water serves as the comparison solution. The calibration curve is made by use of a weakly acidic (H_2SO_4) solution of $Cr_2(SO_4)_3$ containing 0·01 g of Cr. Increasing amounts (0 to 3 ml) of this solution are treated with 1 g of spectroscopically pure Al and the extinction is determined. When the Cr content is as low as 0·001 per cent., the error still does not exceed $\pm$ 2 per cent. Under suitable conditions, solution in alkali can be used, when Cr is obtained quantitatively from the ppt. and can be subsequently determined colorimetrically. H. WREN

622. **Absorptiometric determination of molybdenum in iron and steel.** Methods of Analysis Committee, B.I.S.R.A. (*J. Iron & Steel Inst.*, 1954, **178** [4], 356–359).—The absorptiometric procedure for the determination of molybdenum, based on the formation of a molybdenyl thiocyanate compound, has been critically examined. Modifications have been introduced to suppress, or compensate for, the formation of interfering coloured reaction products. Titanium added to the final solution catalyses the reaction and ensures complete reduction of the ferric thiocyanate. The method has been successfully applied to ferrous materials containing between 0·02 and 5·3 per cent. of molybdenum, particular emphasis being laid on the importance of complete decomposition of the analysis sample. J. O. LAY

623. **Polarographic determination of uranium.** M. V. Šusić (*Bull. Inst. Nuclear Sci., Belgrade*, 1954, **4**, 59–62).—The practicability of determining U in the presence of a number of other ions has been investigated in ascorbic acid, ammonium ascorbate, Na ascorbate, Na phthalate, ammonium phthalate, Na benzoate and ammonium benzoate as supporting electrolytes and a U solution containing Cu, Fe, Ni, Cd, Bi, Sn, Mo, Sb, V, W, Cr, Ag, Pd, Au, As, Ti, Se, Te, Tl and Pb. The results were satisfactory. Te, Tl and Pb, and, in fewer instances, V, Sb, Se and Au, interfered slightly, but interference can be counteracted either by addition of $SnCl_2$, filtration, oxidation of the filtrate by $KMnO_4$ or K_2CrO_4, and recording the U polarogram in ascorbic acid, or by reduction with metallic Cd and recording the U polarogram in any of the above electrolytes except phthalate if the solution contains some of the ions that do not remain in the reductor. L. F. TAYLOR

624. **Polarographic determination of uranium.** D. I. Legge (*Anal. Chem.*, 1954, **26** [10], 1617–1621).—A soln. containing 20 to 1000 μg of U is evaporated to dryness, and the residue is dissolved in 3 *N* HNO_3 (3 ml) in the presence of $Ca(NO_3)_2.6H_2O$ (3 g). The soln. is absorbed in 5 to 10 g of cellulose powder and is then transferred to a column containing 5 g of cellulose powder saturated with nitric acid - ether mixture (5 per cent. v/v of conc. HNO_3 in ether). The eluate (25 ml) and washings (4 × 15-ml portions of nitric acid - ether mixture) are boiled to remove ether, and are then taken to fumes with 3 *M* H_2SO_4 (1 ml), the last traces of organic matter being removed with successive fuming with HNO_3 (0·5 ml) and $HClO_4$ (2 drops). To the cooled residue is added the supporting electrolyte (0·5 *M* oxalic acid containing 0·1 per cent. v/v of conc. HCl, 0·015 per cent. w/v of gelatin as maximum suppressor, and, if necessary, 0·001 per cent. of UO_2^{++} as carrier for very low U concn.) (2·85 ml). The soln. is examined polarographically

under nitrogen between 0 and —0·5 V at 25° ±0·1° C. By comparison with standards, the U concn. is determined with a precision of ± 5 per cent., or more precisely (± 3 per cent.), for > 10^{-4} *M* concn., by direct reading at —0·5 V. Errors from extraneous ions remaining after the cellulose treatment are insignificant. D. A. PANTONY

625. **Rapid determination of uranium in complex minerals such as samarskite, columbite - tantalite and titanoniobates. I. Volumetric determination.** M. M. Tillu (*Proc. Indian Acad. Sci. A.*, 1954, **40** [3], 110–113).—Minerals and alloys of U are extracted with HF, the soln. is reduced with $SnCl_2$ and U is precipitated as the tetrafluoride. Thorium, rare and alkaline earths do not interfere with subsequent titration. *Procedure*—A 5-g sample (containing ≃ 0·2 per cent. of U_3O_8) is evaporated to dryness on a water-bath with 10 ml of 40 per cent. HF, 3 to 4 ml of conc. HCl are added if necessary, and the evaporation is repeated. The residue is treated with 10 ml of HF followed by 2 g of $SnCl_2$, diluted with 50 ml of water, heated on a water-bath for 15 min. and stood overnight at room temp. The soln. is filtered and the residue washed with dil. 40 per cent. HF (1 + 4) and then with water. This residue and the ignited filter-paper are combined in a platinum dish, 2 ml of conc. HNO_3 and 3 ml of conc. H_2SO_4 are added and heat is applied until the mixture fumes. The soln. is then diluted to 50 ml with ice-cold water, passed through a Jones reductor, washed with 2 *N* H_2SO_4 and water, aerated and titrated with 0·05 *N* $KMnO_4$ previously standardised against a similarly treated U soln. If Ce is present, $Ce(SO_4)_2$ is used for the titration. G. R. WHALLEY

626. **Fluorimetric and colorimetric micro-determination of uranium in rocks and minerals.** J. A. S. Adams and W. J. Maeck (*Anal. Chem.*, 1954, **26** [10], 1635–1639).—A sample of uranium ore (1 g) is dissolved either in HNO_3 - HF soln., the F' being removed by successive HNO_3 evaporations, or by refluxing with 50 ml of dil. HNO_3 (1 + 1) for 2 hr. The HNO_3 soln. is filtered and a suitable aliquot of the filtrate is evaporated to dryness. The residue is taken up in warm saturated $Al(NO_3)_3.9H_2O$ (10 ml) that has been extracted with ether to remove U. The U is extracted from the resulting soln. with ethyl acetate (10 ml). Alternatively, the U may be separated by chromatographic treatment on cellulose-powder, by use of 5 per cent. HNO_3 (70 per cent.) in ether as solvent, or the soln. may be treated by a dilution technique. Suitable aliquots of the extract, or eluate and of the standards are heated to dryness and the residues are heated to 660° C with a flux (9 g of NaF fused with 45·5 g of K_2CO_3 and 45·5 g of Na_2CO_3, cooled and ground to pass 80 mesh) (2·5 g) for 30 min. After cooling, the U is determined fluorimetrically by comparison with standards, allowance being made for a reagent blank. Precision is given as ± 7 per cent. For samples containing > 10 p.p.m., a colorimetric procedure is preferred, in which the ethyl acetate extract is treated with an alcoholic soln. of 1:3-diphenylpropanedi-1:3-one, the U concn. being determined at 400 mμ, according to slight modifications of the method given in *Anal. Abstr.*, 1954, **1**, 80. A reproducibility of better than 5 per cent. is claimed. D. A. PANTONY

627. **Halogen determinations by means of adsorption indicators.** G. Müller and A. Detter (*Dtsch. ApothZtg.*, 1954, **94** [46], 1119–1120).—Acid Violet (a triphenylmethane dye) can be used for the quant. determination of Cl and other halogen ions with $AgNO_3$. The Volhard method requires the filtration of silver chloride and back-titration of the excess of silver nitrate; when Acid Violet is used, these procedures are not necessary. The values obtained are accurate and agree with those obtained by the Mohr method and by Fajan's method with fluorescein. Electrolytes do not interfere in the determination, unless they are present in high concn. A. STORFER

628. **The aluminium - morin reaction. II. Colorimetric determination of fluoride ions.** M. T. Beck (*Acta Chim. Hung.*, 1954, **4** [2–4], 223–225.—The colorimetric determination of fluoride ions is based on the observation that they reduce the extinction value of the Al - morin complex. Measurements are effected in ≃ 80 per cent. ethanol, thus giving quick reaction and eliminating the effect of anions often accompanying fluoride ions (NO_3', Cl', Br', I', SO_4''). The calibration curve is made by using 10 ml of a 1·5 × 10^{-4} *M* solution of Al - morin complex, 10 ml of ethanol, and varied amounts of neutral 5 × 10^{-4} *M* KF with water to 25 ml. Experiments are carried out below 15° C on a Pulfrich photometer equipped with an S42 filter. H. WREN

629. **Apparatus for distillation of fluorine as fluorosilicic acid.** R. Mavrodineanu and J. Gwirtsman (*Contr. Boyce Thompson Inst.*, 1954, **17**, 489–494).—An apparatus for the steam-distillation of F (as H_2SiF_6) from a perchloric acid solution is described, in which 500 ml of distillate are collected in 75 min. The electric heating system maintains a temp. of 135° ± 2° C in the distillation flask. The recovery is 98 to 101 per cent. R. H. HURST

630. **Determination of fluorine in glass by the lead chlorofluoride method: application to silicate glasses of high alumina and high boric oxide content.** P. M. C. Proffitt, J. E. Hansen and H. J. Cluley (*J. Soc. Glass Tech.*, 1954, **38**, T277–T284).—For the determination of 0·5 to 2 per cent. of F in glasses containing up to 10 per cent. of Al_2O_3 and 20 per cent. of B_2O_3, the method adopted was that of Hoffman and Lundell (*Bur. Stand. J. Res.*, 1929, **3**, 581), modified to avoid pptn. of $PbCO_3$ and $PbCl_2$ with the PbFCl, which is weighed. *Procedure*—Fuse 1·5 g of sample with 6 g of K_2CO_3 at 900° to 1000° C for 5 min. When cool, digest with 50 ml of hot H_2O for 40 to 60 min. and filter (Whatman No. 41 and 52). Wash with hot H_2O, dilute to 150 ml, stir while adding 2 g of ZnO dissolved in 20 ml of dil. (1 + 4) HNO_3, boil, filter (Whatman No. 44 and 540) and wash. Add to the filtrate (250 ml), aq. NaCl (10 per cent. w/v) (3 ml) and enough dil. (1 + 4) HNO_3 to change bromophenol blue to yellow. Boil to remove CO_2, cool to 70° C, add 2 ml of dil. (1 + 1) HCl and 5 g of $Pb(NO_3)_2$, stir to dissolve, add 5 g of hydrated Na acetate, stir vigorously, digest on a steam-bath for 30 min., set aside overnight, filter on No. 4 sintered-glass crucible, wash, dry at 150° C for 90 min. and weigh. Alternatively, dissolve the final undried ppt. in 50 ml of dil. (1 + 9) HNO_3, dilute to 100 ml, add excess of 0·1 *N* aq. $AgNO_3$, boil to coagulate the AgCl, set aside in darkness for 30 min., filter, wash and titrate the excess of Ag with 0·1 *N* aq. KCNS, using ferric alum as indicator. Recovery of F by this method was 1·05 per cent. for a known fluorine content of 1·11 per cent. J. A. SUGDEN

631. **Estimation of impurities in liquid chlorine by infra-red absorption spectroscopy.** A. W. Pross (*Canad. J. Chem.*, 1954, **32** [10], 956–960).—The determination by infra-red absorption spectroscopy of small amounts of chlorinated organic impurities in liquid Cl is described. The apparatus, including a tantalum-clad steel cell with calcium fluoride windows that are transparent in the infra-red as far as 1180 cm^{-1} and thus exclude the absorption frequencies of liquid Cl, is described and illustrated. Long path lengths increase sensitivity for low concn. of organic compounds and the method is checked by known additions to pure liquid Cl. The determination of moisture in liquid Cl is discussed.
D. Bailey

632. **Stages in the titration of a solution of hydrochloric acid by coulometry at constant intensity.** J. Badoz-Lambling (*Chim. Anal.*, 1954, **36** [11], 291–293).—The titre of HCl solution is determined to an accuracy of 0·2 per cent. by a coulometric method at constant intensity. A 100 per cent. current efficiency is obtained by using a mercury or silver anode of large surface and a platinum cathode dipping directly into the titration solution. Bromophenol blue is used as indicator. The duration of electrolysis ($\simeq$ 10 to 20 min.) gives a measure of the quantity of electricity passed at constant intensity; the current is stabilised to 0·05 per cent. by Sefram, giving absolute values accurate to 0·1 per cent. D. R. Glasson

633. **Catalysed electrode reactions in polarography. II. Polarographic determination of chlorates.** J. Koryta and J. Tenygl (*Chem. Listy*, 1954, **48** [3], 467–469).—In the polarography of the oxalate complex of Ti^{IV} in the presence of ClO_3' ions, the limiting current of $Ti^{\cdots\cdot}$ increases owing to the catalytic reduction of ClO_3'. In a sufficient excess of ClO_3', the limiting catalytic current is proportional to the concn. of $Ti^{\cdots\cdot}$ and to the square root of the concn. of ClO_3'. By means of a theoretical approximation, the concn. ranges in which the limiting current is proportional to the concn. of ClO_3' were calculated. For the determination of ClO_3' in concn. ranges from 0·001 to 0·015 *M*, the following electrolyte is recommended: 0·01 *M* $Ti^{\cdots\cdot}$, 0·2 *M* oxalic acid, 0·4 *M* H_2SO_4, 0·01 per cent. gelatin and 0·25 *M* Na_2SO_4. G. Glaser

634. **Microchemical detection of manganese by diacetylglyoxime thiosemicarbazone.** V. Hovorka and Z. Holzbecher (*Bull. Int. Acad. Tchéque, Sci. Cl. Sci. Math., Natur. et Méd.*, 1953, **51**, 125–126; *Referativnýĭ Zh., Khim.*, 1954, Abstr. No. 20,372).—The addition of an ethanolic soln. of diacetylglyoxime thiosemicarbazone to soln. of Mn salts containing NH_3 produces an intense red-violet colour. For the detection of Mn, paper is treated with the reagent and a drop of test soln. is added; $NH_4^{\cdot}$, $Na^{\cdot}$, $K^{\cdot}$, $Mg^{\cdot\cdot}$, $Ca^{\cdot\cdot}$, $Sr^{\cdot\cdot}$, $Ba^{\cdot\cdot}$, $Sn^{\cdot\cdot\cdot\cdot}$, $Sb^{\cdot\cdot\cdot}$ and $Cr^{\cdot\cdot\cdot}$ do not interfere; Ag, Pb, $Hg^{\cdot\cdot}$, Cu, Cd, Bi, $UO_2^{\cdot\cdot}$, Co and Zn give colours with the reagent. In the presence of tartrate and cyanide, Mn can be detected by a spot test on a white tile even when ions of Ag, Pb, Cu, Cd, Bi, Ni, Zn and Fe^{III} are present. Other metals reduce the sensitivity.
E. Hayes

635. **Photometric determination of manganese with dithiocarbamates.** H. Specker, H. Hartkamp and M. Kuchtner (*Z. anal. Chem.*, 1954, **143** [6], 425–431).—Small amounts (15 to 300 μg) of $Mn^{\cdot\cdot}$ can be determined by oxidation and chelation with Na diethyldithiocarbamate at pH 6·5, extraction with $CHCl_3$ and measurement of the extinction at 578 mμ. Fe, Cu, Co, V, Mo and U are first removed by the extraction of their thiocyanate complexes with a mixture of ether and tetrahydrofuran. The mean error is $\pm$ 2 per cent. A. R. Rogers

636. **Colorimetric determination of traces of iron in metallic copper.** F. G. Zharovskiĭ (*Ukr. Khim. Zh.*, 1953, **19** [5], 548–551; *Referativnýĭ Zh., Khim.*, 1954, Abstr. No. 15,041).—Two methods are described. In the first, the Cu is dissolved in dil. (1 + 1) HNO_3 and, after evaporation and dilution, 0·5 per cent. $MnSO_4$ soln. is added followed by 25 per cent. aq. NH_3 soln. The ppt. of $Fe(OH)_3$ and $Mn(OH)_2$ is dissolved in dil. H_2SO_4 in the presence of KNO_2; after two re-precipitations, the $Fe^{\cdots}$ is determined colorimetrically with KCNS. In the second method, CuCNS is pptd. from the soln., obtained by dissolving the metallic Cu, with KCNS and Na_2SO_3. The soln. is acidified with H_2SO_4 and 2 to 3 drops of $KMnO_4$ are added to oxidise $Fe^{\cdot\cdot}$ to $Fe^{\cdots}$, which is then determined colorimetrically. For an iron content of 0·001 per cent. a 0·2 to 1-g sample is sufficient. E. Hayes

637. **Determination of iron contamination in noble metals.** L. Erdey and É. Bányai (*Acta Chim. Hung.*, 1954, **4** [2–4], 315–324).—Platinum or gold (0·5 g) is dissolved in aqua regia (20 ml) and evaporated to a syrup with conc. HCl (2 ml) soln. and then to dryness, after the addition of NaCl (2 g) and conc. HCl (5 ml). Evaporation to dryness is repeated after the addition of conc. HCl soln. (2 ml). The dry residue is moistened with sufficient conc. HCl to make the solution $\simeq$ 0·1 *N* with respect to HCl when diluted to 100 ml with water. The salt residue is heated at 100° C with a little water until solution is complete and the filtered solution is made up to 100 ml. An aliquot portion, the size of which depends on the expected Fe content, is passed rapidly through a cadmium reductor which is washed repeatedly with water, the washings passing into a 100-ml flask. Either 10 ml of 10 per cent. thioglycollic acid and 10 ml of 10 per cent. aq. NH_3 soln. are added and the solution is made up to 100 ml and the extinction determined with use of an S53 filter, or 5 ml of 40 per cent. sulphosalicylic acid and 20 ml of aq. NH_3 soln. (*d* 0·93) are added, and the solution is made up to 100 ml and observed with the use of an S47 filter. In both cases blank tests are carried out with all the reagents. Up to 0·1 per cent. of Fe in Pt metals can be determined with an error of $\pm$ 5 per cent., or $\pm$ 10 per cent. with an Fe content of 0·1 to 0·01 per cent. Cadmium ions do not interfere. With Ag alloys, reduction is superfluous if the Fe determination is made with sulphosalicylic acid in ammoniacal solution.
H. Wren

638. **Permanent spectrophotometer standard for iron determination.** L. Aconsky, T. Asami and M. Mori (*J. Amer. Wat. Wks. Ass.*, 1954, **46** [9], 894–898).—The use of permanent spectrophotometric standards has eliminated the necessity for preparing fresh standards for each set of iron determinations with KCNS. The permanent standards are made from dilute solutions of potassium dichromate.
A. Webster

639. **Analytical standardisation in the iron and steel industry.** J. O. Lay (*Metallurgia*, 1954, **50** 197–200).—The work of the Methods of Analysis Committee of the British Iron and Steel Research Association in investigating and establishing standard analytical methods is reviewed. G. C. Jones

640. **Studies on organic analytical reagents. V. Ferrous complex salts of α-picolinic acid and quinaldic acid.** K. Shinra, K. Yoshikawa, T. Kato and Y. Nomizo (*J. Chem. Soc. Japan*, 1954, **75** [1], 44).—With the aid of the extinction method, the co-ordination numbers of Fe^{II} complex salts of α-picolinic acid (max. absorption 440 mμ) and quinaldic acid (510 mμ) were found to be 1:3 (Fe to acid) for both. The Fe^{II} complex of α-picolinic acid obeys Beer's law in aq. soln. ($\simeq 10^{-3}$ *M* Fe, pH 7) and can be utilised for the colorimetric determination of Fe in the presence of Cu^{++}, Ni^{++} and Zn^{++} ($\simeq 10^{-3}$ *M*). Quinaldic acid is inferior to picolinic acid because of its sensitivity to light.

K. Saito

641. **Quantitative determination of sodium nitroprusside with sodium nickeltetracyanide.** P. H. Rose (*Z. anal. Chem.*, 1954, **143** [3], 195–198).—The nickel hydroxide formed according to the equation: $4Na_2[Fe(CN)_5]NO + 10NaOH + Na_2Ni(CN)_4 = 4Na_4Fe(CN)_6 + 4NaNO_2 + 4H_2O + Ni(OH)_2$ is estimated with dimethylglyoxime (**I**). The conditions given must be strictly followed to avoid errors caused by co-precipitation and precipitation of ferricyanide and iron hydroxides. *Procedure*—Dissolve 0·1 to 0·5 g of nitroprusside and 0·1 to 0·2 g of $Na_2Ni(CN)_4$ in water (30 ml), add 0·5 g of NaOH and heat to exactly 65° C for 90 min. Filter the blue-green flocculent precipitate, wash it with hot water (150 to 200 ml) and fuse it for 15 min. with Na_2SO_4 and a few drops of conc. H_2SO_4. Cool, add a few drops of conc. H_2SO_4 and fuse. Cool again, dissolve in water and estimate the Ni with **I**.

P. S. Stross

642. **Determination of cobalt in cobalt - nickel solutions.** W. F. Harris and T. R. Sweet (*Anal. Chem.*, 1954, **26** [10], 1648–1649).—The concn. of Co^{++} plus Ni^{++} in a soln. containing Co and Ni only can be determined as follows. *Procedure*—Fifty ml of the soln. are treated with an excess of standard ethylenediaminetetra-acetic acid ($\simeq$ 0·55 per cent., standardised against Zn^{++}), the pH is adjusted to 10 with dil. aq. NH_3 soln. (1 + 1) and the soln. is back-titrated with standard Zn^{++} soln. (0·14 per cent. as ZnO in very dil. HNO_3, standardised gravimetrically as pyrophosphate), with Eriochrome black T as indicator. A second 50-ml aliquot is treated with 10 per cent. HCl (50 ml), and a two-fold excess of 1-nitroso-2-naphthol (6·7 per cent. in glacial acetic acid). After agitation (15 min.), the Co complex is extracted with three 5-ml portions of $CHCl_3$, and the aq. phase is filtered, treated with 30 per cent. H_2O_2 and digested for 20 min. The soln. is cooled, its pH adjusted to 10 with conc. aq. NH_3 soln., and Ni^{II} is determined by titration with ethylenediaminetetra-acetic acid and Zn^{++} as above. The Co^{++} concn. is obtained by difference. Mean deviation is given as 0·25 per cent. for Co and 0·12 per cent. for Ni determinations. The proposed method is applicable to pure Co - Ni soln. only, but Co^{++} may be determined alone if no other ions that react with 1-nitroso-2-naphthol are present.

D. A. Pantony

643. **Volumetric determination of cobalt. Complexiметric titration with ethylenediaminetetra-acetic acid.** W. F. Harris and T. R. Sweet (*Anal. Chem.*, 1954, **26** [10], 1649–1651).—A soln. containing > 50 mg of Co^{++}, and containing no other polyvalent cations, is titrated with excess of standard ethylenediaminetetra-acetic acid. The excess of reagent is back-titrated with standard Zn^{++}, according to details given in *Anal. Abstr.*, 1955, **2**, 642. Precision is claimed to be ± 0·2 per cent.

D. A. Pantony

644. **Studies on metallic complex salts by means of paper chromatography. I. On the nitroammino cobaltic complex salt.** Y. Yamamoto, A. Nakahara and R. Tsuchida (*J. Chem. Soc. Japan*, 1954, **75** [2], 232).—The most satisfactory solvent mixture was composed of 7 vol. of methanol, 2·5 vol. of acetone and 1 vol. of aq. ammonia solution (30 per cent.). The more nitro groups in the ligand, the greater is the R_F value. The *cis*-dinitrotetra-ammino cobaltic complex has bigger R_F values than the corresponding *trans* complex. Mother liquors used in the prep. of $[Co(NH_3)_3(NO_2)_3]$ and $[Co(NH_3)_2(NO_2)_4]K$ were examined by this method. The hexanitro-cobaltic complex appears to be decomposed during the partition process.

K. Saito

645. **The determination of nickel in manganese ores by internal electrolysis.** L. S. Serdyuk and L. U. Barash (*Nauch. Zapisk. Dnepropetrovsk. Gos. Un-ta*, 1953, **43**, 99–104; *Referativnyĭ Zh., Khim.*, 1954, Abstr. No. 15,042).—A rapid method of determining Ni in manganese ores is based on its separation from Mn by internal electrolysis, and subsequent photocolorimetric measurement of the dimethylglyoxime complex, which is oxidised by iodine in alkaline medium. The internal electrolysis is carried out in ammoniacal soln. containing oxalate, which forms a complex with the Mn. The optimum conditions for the separation of Mn and Ni and of the Ni complex are determined.

E. Hayes

646. **The extraction of nickel - dimethylglyoxime with chloroform.** W. Nielsch (*Z. anal. Chem.*, 1954, **143** [4], 272–274).—The extraction of nickel - dimethylglyoxime (Nielsch, *Z. anal. Chem.*, 1953, **140**, 267) can be speeded up by using one chloroform extraction instead of three. The solubility of nickel - dimethylglyoxime in chloroform is such that 1 ml of the saturated solution contains 35 μg of Ni.

P. S. Stross

647. **Organic reagents for the analysis of the platinum metals.** D. E. Ryan (*Analyst*, 1954, **79**, 707–710).—The results are recorded of some preliminary experiments with organic compounds containing nitrogen or sulphur co-ordinating groups, which were examined as possible reagents for the platinum metals. These results indicate the importance of the formation of addition compounds and ammonium-type salts in the analysis. Some reagents worthy of further investigation are described.

A. O. Jones

648. **Chemical analysis of plating solutions.** N. Hall (*Metal Finish.*, 1954, 22nd Ann. Guidebook Directory, 478–494).—Analytical methods requiring simple equipment are given in detail for seventeen plating solutions.

D. J. C. Yates

649. **New internal standard for the quantitative X-ray analysis of shales and mine dusts.** O. G. Griffin (Minist. Fuel Pwr. Safety Min., 1954, Res. Rep. No. 101, 25 pp.).—The new standard [a synthetic form of boehmite (γ AlOOH) prepared by hydrothermal synthesis and available commercially as Cera Hydrate] has been used successfully for over 12 months for the determination of quartz, kaolin and mica in mine dusts, the samples ranging from highly siliceous sandstones to rock dusts of low quartz content. A quartz content of 5 per cent. can be evaluated with an accuracy of ± 15 per cent.

and a quartz content of 2 per cent. to ± 50 per cent.; the uncertainty of a single determination is ≃ ± 10 per cent. Kaolin and quartz exhibit structural variations and the precision is lower for these minerals than for mica. Kaolin can be determined with an accuracy of ≮ ± 15 per cent., the lowest detectable percentage that can be measured being ≃ 5 per cent.; the estimations of kaolin are unaffected by the presence of variable amounts of quartz. The results of applications of the method to the analysis of shale dusts and lung residues are compared with the results of chemical analysis and of X-ray analysis with MgO as internal standard. J. M. JACOBS

650. **Experiments to determine the errors occurring in the preparation of coal samples for laboratory analysis.** R. C. Tomlinson (*J. Inst. Fuel*, 1954, **27**, 515–522).—Experiments on the reduction in size of 50 or 200-lb coal samples are described. Crushing to $\frac{3}{16}$ inch, reduction to 3 lb, crushing to 14 B.S., reduction to $\frac{1}{2}$ lb. and crushing to 72 B.S. gives an accuracy of ± 1 per cent. on coal of < 10 per cent. ash, but less than this at 20 per cent. ash. The error is introduced in the second reduction stage rather than by the size of the original sample. J. G. KING

651. **Systematic analysis of boiler deposits.** J. W. McCoy (*J. Amer. Wat. Wks. Ass.*, 1954, **46** [9], 903–919).—The complete analysis of an internal boiler deposit from the preliminary treatment of the laboratory sample to the final reporting of results is given, and a suggested form of report is contained. Sampling is not considered in this paper; an ion-exchange step has been introduced into the scheme to separate out phosphates and sulphates from the cations. Full working procedures are given. A. WEBSTER

See also Abstracts 672, 695, 696, 697, 757, 758, 769, 770, 794.

3.—ORGANIC ANALYSIS

652. **The development of organic elementary analysis.** A. A. van der Wal (*Chem. Weekbl.*, 1954, **50** [48], 829–835).—An historical account of the development of organic elementary analysis is given. E. HAYES

653. **A modified apparatus for use with the rapid micro Dumas procedure.** H. Swift (*Analyst*, 1954, **79**, 718–721).—The apparatus previously described (Swift *et al. Brit. Abstr. C*, 1952, 579) for the rapid determination of N in organic compounds by combustion is modified to include a flowmeter that could, with the minimum of adjustment, be readily freed from air. The gas flow is constricted by the sliding-rod principle of White *et al.* (*Brit. Abstr. A II*, 1937, 128), and dibutyl phthalate coloured with Sudan red serves as the indicator liquid. The construction and manipulation of the modified apparatus are described. A. O. JONES

654. **A new method for ultramicro-determination of ammonia or organic nitrogen.** Y. Okada and H. Hanafusa (*Bull. Chem. Soc. Japan*, 1954, **27** [7], 478).—A micro-Kjeldahl method, modified for determining 30 μg of nitrogen, is reported. Colorimetry with ninhydrin is used, the sample for a single determination contains 20 to 100 μg of nitrogen, the time of distillation is 1·5 min., and 5 ml of 0·01 *N* H_2SO_4 soln. are placed in an adapting flask. After distillation, the soln. is diluted with a citrate buffer (pH 5·0) and a 1-ml aliquot is taken for colorimetric determination of NH_3. A standard curve is prepared previously with a soln. of NH_4Cl (1 to 10 μg per ml). Results for total nitrogen in acetanilide, and of amide ammonia in lysozyme and Taka-amylase A are reported. O. M. WHITTON

655. **Semi-micro method of determining sulphur in organic compounds.** A. G. Yudasina and L. D. Vÿsochina (*Nauch. Zapisk. Dnepropetrovsk. Gos. Un-ta*, 1953, **43**, 53–56; *Referativnÿĭ Zh., Khim.*, 1954, Abstr. No. 15,061).—The compound is hydrogenated in a porcelain tube containing a platinum spiral. The tube is heated to 850° C in an electric furnace and the H_2S produced by 30 to 40 mg of sample is absorbed in a mixture of 10 ml of 10 per cent. $ZnSO_4$ soln., 10 ml of 10 per cent. Na acetate soln. and 1 ml of glacial acetic acid. On completion of the decomposition, the contents of the receiver are mixed with acid 0·02 *N* iodine soln. and the excess of iodine is titrated with 0·02 *N* $Na_2S_2O_3$. With slight modifications to the apparatus, the method can be adapted to micro-determinations. The method is not applicable to compounds containing phosphorus because of catalyst poisoning. The limits of error are ± 0·2 per cent. E. HAYES

656. **Absorption of oxides of sulphur by metallic oxides and silicates.** P. N. Fedoseev and R. M. Lagoshnaya (*Zh. Anal. Khim., SSSR*, 1954, **9** [4], 224–228).—To determine S by combustion, the organic substance is heated at 800° C in an empty tube in the presence of Cr_2O_3 as catalyst; the sulphur oxides are absorbed at 700° to 800° C by solid absorbents in boats placed in the combustion tube. The rate of flow of O is 50 ml per min. Of various oxides and silicates tested as absorbents, sodium silicate is recommended. G. S. SMITH

657. **A critical study of the determination of methoxyl groups.** A. E. Heron, R. H. Reed, H. E. Stagg and H. Watson (*Analyst*, 1954, **79**, 671–680).—The cause of some erratic results obtained when the method of Easterbrook *et al.* (*Brit. Abstr. C*, 1953, 505) for the determination of methoxyl groups in cellulose esters was applied to pure methoxy compounds was traced to the $Na_2S_2O_3$ in the wash soln. used to remove HI from the stream of gas carrying methyl iodide into the absorber. A 25 per cent. aq. sodium acetate soln. is a satisfactory wash soln. The slightly modified Easterbrook apparatus is described and illustrated, and the procedures for micro and semi-micro determinations are given. The average recovery from vanillin and α-methyl-D-glucoside is 100 per cent. with a coefficient of variation of 0·21. A. O. JONES

658. **Observations on the detection of mercapto groups.** H. Freytag (*Z. anal. Chem.*, 1954, **143** [6], 401–414).—Methods involving ferric ferricyanide, cupric ferricyanide, 2:3:5-triphenyltetrazolium chloride, dimethyl-*p*-phenylenediamine and $FeCl_3$ or brucine and potassium persulphate for the detection of cysteine, cystine or thioglycollic acid in biological material are critically examined. A. R. ROGERS

659. **Infra-red spectroscopy as aid in quantitative analysis of hydrocarbon mixtures.** W. Fuchs and H. Pietsch (*Erdöl u. Kohle*, 1954, **7** [10], 631–632).—Extinctions at 9·0 and 9·2 μ measured by a semi-automatic spectrometer with a rock-salt prism gave

values for mole-fractions of *n*-hexane in mixtures with toluene; there was an average error of + 1·7 per cent. For the ternary mixture with benzene, measurements at 5·3, 9·0 and 11·25 μ gave an average error of + 1·3 per cent. A determinant for solving the three equations involved is tabulated.
A. R. PEARSON

660. **Indicator chromatographic analysis of organic mixtures.** H. S. Knight and S. Groennings (*Anal. Chem.*, 1954, **26** [10], 1549–1553).—Indicator adsorption methods are described for the analysis of mixtures of polar organic liquids such as alcohols, ketones and amines. The eluotropic series, or order of adsorbability, of the components of each system was determined and suitable oil-soluble dyes were selected to serve as indicators. In non-ideal systems the sample is abnormally adsorbed and may be by-passed by the eluent; techniques for utilising or avoiding by-passing by the use of special eluents are described. A few systems were analysed by frontal analysis in which a larger sample is forced through the column without the use of a developer; at the liquid front the most weakly adsorbed component forms a zone the length of which is indicated by the appropriate dye. A qual. theory of indicator analysis to explain the observed phenomena is presented. The accuracy of the methods varies from ± 0·1 to ± 4·0 per cent., depending on the system used.
G. P. COOK

661. **Characterisation of the lower alcohols (C_1 to C_5) by the eutectic points of their xanthates with dicyanodiamide.** W. Antoni and E. Appel (*Z. anal. Chem.*, 1954, **143** [2], 114–117).—The method of Whitmore and Lieber (*Ind. Eng. Chem., Anal. Ed.*, 1935, **7**, 127) is modified. Instead of determining the melting points of the xanthates of the alcohols, their eutectic points with dicyanodiamide are determined. These are more reproducible and less affected by decomposition. The eutectic points of the normal C_1 to C_4 alcohols differ by about 10° C. *n*-Propanol and *iso*propanol are easily distinguished, but the isomers of butanol and pentanol are not.
P. S. STROSS

662. **Determination of small amounts of ketones by ultra-violet colorimetry.** S. A. Shchukarev, S. N. Andreev and O. V. Sapozhnikova (*Zh. Anal. Khim., SSSR*, 1954, **9** [4], 193–195).—Ultra-violet absorptiometry (*Brit. Abstr. C*, 1953, 407) is applied to the determination of acetone, ethyl methyl ketone, pentan-2-one, hexan-2-one, heptan-2-one and octan-2-one by the iodoform reaction; the absorptiometer described previously (Andreev, *Brit. Abstr. C*, 1953, 35) is used.
G. S. SMITH

663. **Micro-titration of organic compounds [acids]. I. Semi-automatic apparatus for micro-titration.** W. Simon, E. Kovák, L. H. Chopard-dit-Jean and E. Heilbronner (*Helv. Chim. Acta*, 1954, **37** [6], 1872–1885).—A semi-automatic titration apparatus is shown and described with which micro-titrations, on ≃ 1 mg of a compound of equiv. wt. 300, may be carried out. A statistical analysis, with benzoic acid as the test substance, has been carried out with the following results concerning the accuracy of single determinations on the 95 per cent. confidence limit: pK ± 0·07 units, equiv. wt. ± 4 per cent.
H. WREN

664. **Volumetric method for the analysis of acetic acid derivatives in mixtures from industrial syntheses.** C. Capitani and P. Imperiale (*Chim. e Ind.*, 1954, **36** [8], 606–611).—Methods for the determination of acetic acid, acetic anhydride, vinyl acetate, ethylidene diacetate and acetaldehyde in the presence of one another, are described. Acetic acid is determined by titration at 0°C with aq. NH_3 solution and use of a neutral red - methylene blue indicator changing from violet to green at pH 7. The other components determined by standard methods are: acetic anhydride, by reaction with excess of aniline in chlorobenzene solution and titration of unreacted aniline with $HClO_4$; vinyl acetate, by titration in aq. acid solution at 0° C with bromide - bromate, by use of a dead-stop indicator; acetaldehyde, by a bisulphite method; and ethylidene diacetate by hot saponification with NaOH soln., with allowance for the acetic acid, acetic anhydride and vinyl acetate (which also react). Results obtained on an artificial mixture of all components and on an industrial reaction mixture are reported.
L. A. O'NEILL

665. **Permanganate oxidation of malic acid and its application to quantitative analysis.** J. F. Casas Lucas and J. Arnedo Rodríguez (*An. Soc. Esp. Fis. Quím., B*, 1954, **50** [9–10], 737–740).—The method of Peynaud (*Ann. Falsif.*, 1938, **31**, 332) is investigated, for variations of the pH. The acetaldehyde liberated is measured by one of the techniques described in an earlier paper (Casas Lucas, *An. Soc. Esp. Fis. Quím., B*, 1954, **50**, 535). The limits of pH for accuracy are 5·0 to 5·4, and the method is accurate to within 1 per cent. for 1·34 to 16-mg samples of malic acid.
D. LEIGHTON

666. **Qualitative differentiation between certain O-methyl derivatives of D-fructose using borate-paper electrophoresis and the urea - hydrochloric acid spray.** D. J. Bell and D. H. Northcote (*Chem. & Ind.*, 1954, [43], 1328).—A number of O-methyl derivatives of D-fructose can be readily differentiated by borate-paper electrophoresis. With a urea - hydrochloric acid spray, to reveal the sugar spots, not all the substances give the green-grey colour exhibited by fructose and its 1:3:4:6-tetra-, 1:3:4-tri- and 3:4-di-O-methyl derivatives. The methylfructoses which are fixed in the pyranose form all give a brown-ochre colour with the spray, but in the presence of borate a brown tinge is imparted to spots of free fructose, sucrose and methyl β-D-fructopyranoside in distinction to the grey-green colour found in the absence of borate. It is suggested that the fructose molecules formed in the presence of the acid spray become fixed in the pyranose form by coupling with borate through hydroxyls 4 and 5. The structure of the borate complexes is discussed and experimental results for the migration of the methyl fructoses are given. From the results, it is seen that the method provides an analytical procedure for the separation of these derivatives, although their configuration and possible complex formation with borate cannot be directly correlated with their speeds of migration.
A. LEDWITH

667. **The quantitative determination of *p*-hydroxyphenylpropionic acid.** A. K. Ruzhentseva and N. S. Goryacheva (*Aptechnoe Delo*, 1954, **3** [5], 7–8).—*p*-Hydroxyphenylpropionic acid is determined by bromination at 0°C for 3 min., when it is quant. converted to the 3:5-dibromo derivative. Dissolve the sample (0·09 to 0·1 g) in 10 ml of water in a 300-ml flask by gentle heating. To the cooled solution, add 40 ml of 0·1 *N* $KBrO_3$ soln. and 10 ml of 20 per cent. KBr soln. Set aside the stoppered flask in an ice-bath for 10 min. Add 15 ml of

ice-cooled 20 per cent. H_2SO_4 and mix thoroughly; after exactly 3 min., add 25 ml of 20 per cent. KI soln. Remove from the ice-bath and titrate the liberated iodine with 0·1 *N* $Na_2S_2O_3$. Higher temp. or increase in bromination time results in further bromination; at 20° to 22° C and with a reaction time of 30 to 40 min., 3 atoms of Br are taken up.

E. HAYES

668. **The photometric determination of quaternary ammonium salts and of certain amines by compound formation with indicators. I. Quaternary ammonium salts.** C. W. Ballard, J. Isaacs and P. G. W. Scott (*J. Pharm. Pharmacol.*, 1954, **6** [12], 971–984).—Published methods for the determination of quaternary ammonium salts by titration with anionic surface-active agents, and by measuring the change in colour intensity produced in an indicator in aq. soln., are not applicable to salts of mol. wt. lower than that of cetrimide. The effects of varying the conditions in the indicator extraction method have been studied, and the following general method is proposed. *Procedure*—To a soln. (5 ml, approx. 0·001 *M*) of the quaternary ammonium salt, add 0·15 per cent. bromothymol blue soln. (1 ml) and citrate - phosphate buffer at pH 7·5 (20 ml). Extract with chloroform B.P. (3 × 20 ml). Shake the bulked extracts for 1 min. with glass wool (0·4 g); after 5 min., decant through a glass-wool plug into a 100-ml calibrated flask containing a solution of boric acid (0·5 g) in 90 per cent. v/v ethanol (25 ml). Dilute to vol. with chloroform, and measure the extinction of 2 cm at 420 mμ against chloroform. Calculate results from a standard curve. The method is applicable to all quaternary salts except those of very low mol. wt. (such as choline). Preliminary results on the effect of interfering substances are given.

A. R. ROGERS

669. **The optical properties of L-cystine.** R. Maurenbrecher (*Z. anal. Chem.*, 1954, **143** [3], 176–182).—A study of the specific rotation of L-cystine under various conditions showed that it falls rapidly with a rise in temperature in acid solutions (*e.g.*, $[\alpha]_D^{8°\,C} = -253°$, and $[\alpha]_D^{32°\,C} = -197°$). Solvent concentration also has considerable influence, and curves for specific rotation in various concn. of solvents such as H_2SO_4, HCl and NaOH are given. The concentration of cystine has little effect; similar effects have been found with other amino-acids such as tyrosine and leucine.

P. S. STROSS

670. **Displacement of the nitro-group during analysis of nitrophenols and nitroanilines by the Koppeschaar method.** L. D. Johnson (*Dissert. Abstr.*, 1954, **14** [9], 1304).—When applying the Koppeschaar bromination procedure to nitrophenols or nitroanilines, an excess of aq. Br may (through the action of free Br or HOBr) cause displacement of the nitro group (especially when in the *o*- or *p*-position) with a resultant consumption of Br greater than the theoretical amount and a fugitive end-point in the titration with $Na_2S_2O_3$. Displacement increases with temp. and time of bromination, but is retarded by increased acidity of the mixture and by operating at 0° to 10° C; normal bromination is complete in 5 to 10 min. The displacement reaction is complex, but probably produces HNO_3 initially and then HNO_2 by secondary reactions; the system eventually attains equilibrium. A method for determining 2:4-dinitrophenol and picric acid, when present together, is described. The Koppeschaar procedure is inapplicable quant. to those nitrophenols, *e.g.*, nitroresorcinols, that consume large amounts of Br.

W. J. BAKER

671. **Colorimetric determination of 2-bromo-1-methoxy-2-nitro-1-phenylpropane.** L. R. Jones and J. A. Riddick (*J. Amer. Pharm. Ass., Sci. Ed.*, 1954, **43** [11], 679–680).—To a solution of 2-bromo-1-methoxy-2-nitro-1-phenylpropane (**I**) (≃ 1 mg) in butanol (5 ml), add 0·5 *N* methanolic NaOH (1 ml). Heat for 10 min. on a steam-bath and cool. Add a mixture of 0·5 per cent. $FeCl_3.6H_2O$ (2 ml) and 0·6 *N* methanolic HCl (1 ml), and dilute to 12·5 ml with ethanol. Measure the extinction at 490 mμ in a 1-cm cell against a blank prepared in the same way but without heating for 10 min. For the assay of ointments containing 0·5 to 1·0 per cent. of **I**, extract the **I** either with butanol or with *n*-hexane and methyl cyanide, evaporate and dissolve the residue in butanol. The method is rapid and has an accuracy of ≃ 5 per cent. and a precision of ≃ 2 per cent.

A. R. ROGERS

672. **Colour reactions of some mercurated arylamines with nitrites.** I. M. Korenman and A. A. Belyakov (*Zh. Anal. Khim., SSSR*, 1954, **9** [4], 220–223).—Mercuration of secondary and tertiary arylamines increases the sensitivity of the colorimetric reaction with nitrites by 2·5 to 16 times. To determine nitrite, 4 ml of the solution are mixed with 0·1 ml of reagent (10 mg per ml of an organomercury compound, *e.g.*, *p*-dimethylaminophenylmercury acetate, in 10 per cent. acetic acid), and 1 ml of 1 per cent. acetic acid; the mixture is heated at 85° to 100° C for 5 min., and then cooled and treated with 0·1 ml of 20 per cent. Na acetate solution. The colour is compared with a series of standards containing 0·1 to 20 μg of NO_2' in 5 ml. The Hg compound can be made as required, *e.g.*, by heating 20 mg of Hg acetate with 6 mg of dimethylaniline in 1 ml of 1 per cent. acetic acid. To determine arylamines, 4 ml of the solution in ethanol, acetone or aniline are mixed with 0·1 ml of Hg acetate solution (15 mg per ml in 1 per cent. acetic acid), heated to 85° to 100° C and treated with 0·1 ml of 1 per cent. $NaNO_2$ solution for 5 min. After cooling, 0·4 ml of 20 per cent. Na acetate solution is added, and the colour is compared with a series of standards containing 0·5 to 25 μg of tertiary, or 4 to 200 μg of secondary, arylamine in 5 ml of acetic acid.

G. S. SMITH

673. **Aromatic diazo compounds. XVI. Determination of 6-amino-1-naphthol-3-sulphonic acid (J-acid).** M. Matrka, J. Sova and Z. J. Allan (*Chem. Listy*, 1954, **48** [3], 417–420).—The existing methods for the determination of J-acid are re-examined and a new method is proposed, based on the coupling of J-acid (0·005 mole in 5 ml of 2·5 *N* Na_2CO_3) with 0·1 *N* *p*-nitrophenyldiazonium chloride in a saturated soln. of sodium xylenesulphonate (150 ml) and 2·5 *N* HCl (10 ml) at 5° C. To detect the presence of γ-acid (7-amino-1-naphthol-3-sulphonic acid) in technical J-acid, carry out the coupling in weakly acidic (acetic acid) aq. soln., destroy the excess of the diazonium salt by the addition of resorcinol, dilute the mixture, treat it, after warming, with an aq. NH_3 soln. and chromatograph on paper with 20 per cent. aq. NH_3; the appearance of a single characteristic violet spot is due to γ-acid.

G. GLASER

674. **The detection and differentiation of some aromatic amines.** M. Z. Barakat, N. Wahba and M. M. El-Sadr (*Analyst*, 1954, **79**, 715–717).—A test to distinguish by colour reaction between primary, secondary and tertiary aromatic amines, or between various members of these series, is proposed. The test depends upon the colour developed when an aromatic amine reacts with N-bromosuccinimide in an aq. medium. The coloured compound is easily extracted from the aq. phase with $CHCl_3$. The same test, with subsequent addition of conc. H_2SO_4, differentiates between primary amines and also the toluidines. New colour tests to differentiate between aromatic amines with cyclic triketones, *viz.*, ninhydrin, *peri*naphthindanetrione hydrate, *m*-nitro*peri*naphthindanetrione hydrate and alloxan, are described. The colours given by various amines with these reagents are tabulated. A. O. JONES

675. **4-*n*-Octyloxyphenylguanidine chloride; a new reagent for the quantitative determination of picrate ions.** C. Pasini and A. Vercellone (*Z. anal. Chem.*, 1954, **143** [3], 172–176).—Increasing numbers of C atoms in the alkyl radicle decrease the solubility of the picrates of the 4-alkoxyphenylguanidine salts. The octyloxy compound is suitable for the gravimetric determination of picrate ions between pH 2·5 and 10·5. *Procedure*—To a solution, at a temperature of 40° to 60° C, containing picrate ions (≃ equivalent to 5 ml of 0·01 *N*), add the 4-octyloxyphenylguanidine chloride reagent (**I**) (15 ml) (0·2944 g of **I** dissolved in 20 to 25 ml of hot 0·05 *N* HCl and made up to 100 ml), stir and allow to stand for several hours. Filter, wash and dry the precipitate for 1 hr. at 105° C. Dissolve the precipitate in acetone, and evaporate the acetone solution to dryness in a tared flask. Dry the residue at 105° to 110° C to constant weight. 1 g of precipitate ≡ 0·465 g of picric acid. P. S. STROSS

676. **Colour reactions of carbazole, indole, pyrrole and some of their derivatives with bromonitroindanedione.** G. Vanag (*Zh. Anal. Khim., SSSR*, 1954, **9** [4], 217–219).—In glacial acetic acid 2-bromo-2-nitroindanedi-1:3-one gives red, violet or blue colours with carbazole, indole, pyrrole and some of their derivatives. Tryptophan gives a pink colour with a red-green fluorescence. All other amino-acids tested and some of the derivatives of carbazole, indole and pyrrole give a yellow colour. The limiting dilution for the reaction with carbazole is 1 in 500,000. G. S. SMITH

677. **The fluorescence of quinoline in acid solutions.** S. Šljivić (*Z. anal. Chem.*, 1954, **143** [2], 113–114).—Solutions of quinoline absorb light in the u.v. and emit between 425 and 565 mμ. The width of the band and the intensity depend on the concn. of the acid present; in conc. HNO_3 and conc. H_2SO_4, light of longer wavelengths is emitted. In general, conc. acids or acid solutions of NaCl, KBr and KI decrease the intensity of fluorescence. No noticeable decrease is caused by HF. As alkaline solutions do not fluoresce, the uses of quinoline as a fluorescent indicator are being studied. P. S. STROSS

678. **Amperometric titration of 8-hydroxyquinoline and some derivatives with potassium bromate.** Q. Fernando (*Analyst*, 1954, **79**, 713–714).—The amperometric titration of 8-hydroxyquinoline (**I**), 8-hydroxyquinaldine (**II**) and 8-hydroxyquinoline-5-sulphonic acid (**III**) with $KBrO_3$ is described. In a 3×10^{-4} *M* soln. these can be determined with an accuracy of ≃ 1 per cent.; in a 2×10^{-5} *M* soln., **I** and **II** can be determined with an accuracy of ≃ 5 per cent. and in a 9×10^{-5} *M* soln., **III** can be determined with an accuracy of ≃ 3 per cent. A S.C.E. and a mercury-sealed rotating platinum micro-electrode similar to that described by Laitenen and Kolthoff (*J. Phys. Chem.*, 1941, **45**, 1079) were used. A. O. JONES

679. **Quantitative infra-red spectroscopic analysis of α-, β- and γ-picolines and 2:6-lutidine.** C. A. Ceselli (*Chim. e Ind.*, 1954, **36** [8], 595–598).—Specific extinction coefficients (in CS_2 solution) are determined at 12·57μ, 12·76μ, 12·84μ and 13·32μ for γ- and β-picoline, 2:6-lutidine and α-picoline, respectively. Agreement with Beer's law is not shown, so the specific extinction coefficient of the test solution must be compared with that of a solution of the pure compound at an approx. similar concn. Analyses of fifteen synthetic mixtures are shown, results being accurate to within 2 per cent. for all components. L. A. O'NEILL

680. **Qualitative analysis of silicon-organic compounds by the method of infra-red absorption spectroscopy.** A. P. Kreshkov, Yu. Ya. Mikhallenko and G. F. Yakimovich (*Zh. Anal. Khim., SSSR*, 1954, **9** [4], 208–216).—Diagrams are given of the infra-red absorption spectra of eleven silicon-organic compounds between 2 and 10 μ. Wavelengths representative of various groupings are assigned, *viz.*, CH_3·, 3·45 and 6·8 to 7·1; C_6H_5·, 3·25, 6·25, 6·7 and 9·7; ⋮Si·C⋮, 7·95 to 8·0, ⋮Si·O· 9·5 to 9·65; ·O·R 8·6 to 8·7; and ·CH:CH_2, 6·15 μ. G. S. SMITH

681. **The chromatographic separation of crude benzene and its commercial fractions.** D. G. Yurko (*Nauch. Zapisk. Dnepropetrovsk. Gos. Un-ta*, 1953, **43**, 57–65; *Referativnyĭ Zh., Khim.*, 1954, Abstr. No. 15,076).—Aromatic hydrocarbons are separated by liquid chromatography on silica gel. With water at 18° to 23° C as the displacer, the hydrocarbons are adsorbed in the order: *o*-xylene, benzene, *m*-xylene, toluene, ethylbenzene and *p*-xylene. Benzene is more strongly adsorbed than thiophen and sulphur compounds, and it can be separated from mixtures with these substances. The polynitro derivatives of aromatic hydrocarbons, and their coloured forms obtained by the action of KOH, can be separated on alumina columns from a CCl_4 soln. E. HAVES

682. **The determination of thiophen in commercial benzene.** Z. F. Solomko (*Nauch. Zap. Dnepropetrovskogo Gos. Un-ta*, 1953, **43**, 37–44; *Referativnyĭ Zh., Khim.*, 1954, Abstr. No. 16854).—For the determination of 0·0005 to 0·01 per cent. of thiophen in benzene, its reaction with isatin in the presence of $Fe_2(SO_4)_3$ is used. The reagent [0·01 g of isatin and 0·01 g of $Fe_2(SO_4)_3$ in 100 ml of conc. H_2SO_4] is stabilised by heating on a bath of boiling water for 15 to 20 min. The reagent (3 ml) is mixed with the benzene sample for one minute and after 6 to 8 min. a stable colour is obtained. Up to 0·5 per cent. of CS_2 does not interfere. The relative error is ± 5 per cent. E. HAYES

683. **The determination of carbon disulphide in commercial and crude benzene.** F. I. Berezovskaya and Z. F. Solomko (*Nauch. Zap. Dnepropetrovskogo Gos. Un-ta*, 1953, **43**, 31–35; *Referativnyĭ Zh., Khim.*, 1954, Abstr. No. 16,829).—Carbon disulphide

is converted to the ethylxanthate, and the red-violet colour of the Mo complex, which this compound forms in acid medium, is measured colorimetrically. *Procedure*—Stir 2·5 ml of the benzene for 5 min. with 15 ml of ethanolic alkali, distil off the benzene and ethanol, and dilute the residue to 250 ml with water. To 0·5 ml of this soln., add 1·5 ml of 15 per cent. ammonium molybdate, 1·2 ml of 10 per cent. HCl and water to 10 ml, mix and determine the intensity of colour in a photocolorimeter. Read the CS_2 content from a calibration curve. The sensitivity limit is 0·001 mg per ml. E. HAYES

684. **Quantitative determination of vat dyes on wool fibre.** N. G. Klemin, P. V. Moryganov and A. A. Solov'ev (*Zh. Prikl. Khim., SSSR*, 1954, **27** [7], 797–800).—About 0·1 g of the fabric is treated with 10 ml of conc. H_2SO_4 (*d* 1·84) at 110° C for 5 min. The mixture is cooled and transferred carefully into 0·1 per cent. gelatin solution in a 100-ml calibrated flask, and made up to the mark with the same solution. The colour of this solution is compared against a series of standards prepared in the same way with known amounts of dye and undyed fibre. The results are reproducible to within 5 per cent. and more accurate than those given by other methods. R. C. MURRAY

685. **Potentiometric research on viscose solutions. II. Communication on viscose analysis.** B. Phillipp (*Faserforsch. u. Textiltech.*, 1954, **5** [9], 337–347).—A review is given of the earlier work on the analysis of viscose, by potentiometric titration with $HgCl_2$ and with $AgNO_3$, to determine consecutively the sulphide (**I**), thiocarbonate (**II**) and xanthate (**III**). A method of rapid analysis has been devised that is suitable for series analysis and takes 1½ to 2 hr. to complete. In this method **I**, **II** and **III** are precipitated consecutively by titration with 0·1 *N* $AgNO_3$ soln. from a microburette. The potential measurement is effected with a rotating Ag electrode against a calomel electrode. A weighed (≃ 2-g) sample of viscose is mixed with 30 ml of water, then with 30 ml of 2 *N* Na acetate solution (for buffering in subsequent neutralisation), and finally covered with a layer of liquid paraffin to exclude air (and avoid errors through action of the air on the S compounds). **I** is pptd. at pH 12 to 13, then the solution is carefully neutralised to pH 8 with 0·5 *N* acetic acid, added drop by drop and with suspension of the stirring to avoid loss of H_2S; then **II** is pptd. After removing the Ag_2S coating from the electrode, titration is continued to ppt. **III**. The results for **III** agree with the iodimetric gamma values but the total values found for **I**, **II** and **III** are 5 to 10 per cent. below those found gravimetrically. It has yet to be established whether this is due to the presence of further S compounds or whether both the method and the determination of the gamma values give too low a figure for **III**. The ideas of Herrent on the after-ripening (stagewise decomp. of xanthate) could not be confirmed, but his variation of the potential course of the silver-cell xanthate precipitation with the time of after-ripening of the viscose is established. H. L. WHITEHEAD

686. **Estimation of the intrinsic viscosity of polyethylene terephthalate solutions.** H. Frind (*Faserforsch. u. Textiltech.*, 1954, **5**, 290–296).—A (1 + 1) mixture of phenol and tetrachloroethane (**I**) is found to be a suitable solvent for the determination of the fundamental or intrinsic viscosity (η) of polyethylene terephthalate (**II**); a mixture of phenol and water is unsuitable. Minor variations of the ratio of phenol to **I** do not affect the results. The relative viscosities ($\eta_{rel.}$) of a series of solutions of 4 samples of **II** of concn. 2·0 to 0·04 g per 100 ml are measured at 20° ± 0·005° C, using a Ubbelohde viscometer. The solutions used are prepared by treating **II** (2 g of low mol. wt., or 1 g of high mol. wt.) with ≃ 25 ml of the solvent, swelling at room temp. overnight, and dissolving at 80° C with addition of portions of solvent and making up to 100 ml. The $\eta_{rel.}$ is obtained from the equation $\eta_{rel.} = \frac{t}{t_0}$, where t and t_0 are, respectively, the times of flow of the solution and of the solvent, after correction by the Hagenbach factor for kinetic energy and ignoring the difference of density. From $\eta_{rel.}$ the specific viscosity ($\eta_{sp.}$) is obtained from $\eta_{sp.} = \eta_{rel.} - 1$. In graphs of $\frac{\eta_{sp.}}{c} = f(c)$ (where c is the concn.), especially with the lowest mol. wt. polymers, the graph passes through a minimum at ≃ 0·1 g per 100 ml and then becomes linear with the increase in concn. The reasons for this abnormal rise with low concn. are discussed. The linear rise, which describes the Huggins' equation $\frac{\eta_{sp.}}{c} = \eta + k(\eta)^2 c$ (where k is 0·35), is used to obtain η by a linear mathematical extrapolation. By transforming that equation to $\eta = \frac{\sqrt{1 + 1{\cdot}4\ \eta_{sp.}} - 1}{0{\cdot}7\ c}$, η is also calculated from measurement of $\eta_{sp.}$ for any concn. The calculated values and the extrapolated values vary on an average by ± 1 per cent. H. L. WHITEHEAD

687. **Photocolorimetric examination of colours.** J. E. Goris (*Chim. Peint.*, 1954, **17** [7], 217–220).—The use of photocolorimeters for the study of colour problems is described. L. A. O'NEILL

688. **Analysis of butylated hydroxyanisole in paper and paperboard.** J. J. Austin (*J. Amer. Oil Chem. Soc.*, 1954, **31** [10], 424–427).—Qual. and quant. methods for the determination of butylated hydroxyanisole (BHA) in paper and paperboard are given. BHA is extracted from paper or paperboard with 80 per cent. ethanol for 10 hr. in a Soxhlet and estimated by a colorimetric technique with a 0·1 per cent. solution of 2:2′-dipyridyl in absolute ethanol and a 0·0832 per cent. solution of ferric chloride hexahydrate in absolute ethanol. Before measurement of the colour, 30 min. ageing time is required. A new rapid method for the estimation of BHA in paper only, that can be completed in 25 min., is described. The method is based on the colour reaction between 2:6-dichloroquinonechlorimine (2:6-D) and phenols; the colour reaction is sensitive to pH, giving max. colour at pH 9·4 (borax buffer), when the colour formed is stable for at least 5 hr. Maximum colour intensity is formed within 15 min. after the addition of the reagents. The latter method is also used as a qual. test for the detection of BHA in paper and to determine which side of the paper is treated. I. JONES

689. **Assay of greases. Determination of melting [flowing] point and dropping point.** German Standard DIN 51801 (*Erdöl u. Kohle*, 1954, **7** [10], 659–660).—The apparatus consists of a closed-scale thermometer fitted with two cylindrical metal

hulls and a metal nipple at the lower end. The flowing point is the temp. at which the grease forms a complete hemispherical globule under the nipple; the dropping point is the temp. at which the first drop of grease falls from the nipple. The temp. is raised at a rate of about 1° C per min.; the results are accurate to ± 4° C. D. R. GLASSON

690. **Assay of greases. Determination of ionisable chlorine content.** German Standard DIN 51800 (*Erdöl u. Kohle*, 1954, **7** [10], 660).—The grease (100 g) is boiled with distilled water (200 to 300 ml) and HNO_3 (sp. gr. 1·40) (15 ml) until the aq. solution is clear. After treatment with paraffin wax (fully refined, m.p. 52° to 54° C) followed by filtration, the Cl is determined by Volhard's method; results are accurate to ± 0·002 per cent. of Cl. D. R. GLASSON

691. **Radio-active isotopes in paint technology.** G. L. Vandone (*Pitture e Vern.*, 1954, **10** [10], 639–645).—This account of the properties of radio-active isotopes and their uses in analysis is followed by a discussion of possible applications in paint technology, *e.g.*, in the radiography of paint films to detect flaws, in the determination of film thickness, as tracers, and in studies of water-permeability and plasticiser migration. D. R. DUNCAN

692. **Analysis of varnish vehicles. III. Effect of cooking on oil-resin systems.** R. Montequi and J. López Morales (*An. Soc. Esp. Fis. Quim., B*, 1954, **50** [6], 561–564).—The effect of cooking for 3 hr. at 290° C on the acid val., sap. val. (under mild and strong conditions), I val. and η of mixtures of linseed oil with rosin, Ca or Zn resinate, ester gum and phenolic and coumarone resins has been determined, to assist in the interpretation of results obtained in the analysis of varnishes. Except in the case of reactive phenolic resins, the main changes on cooking can be related to the polymerisation of the oil.
L. A. O'NEILL

693. **The determination of the exact molecular weight of macro-molecules from rheological measurements. Rheology of solutions of high polymers. VI.** K. Edelmann (*Fraserforsch. u. Textiltech.*, 1954, **5** [8], 325–337).—The problem of determining the exact mol. wt. and the degree of polymerisation of macro-molecules by classic and thermodynamic methods is discussed; methods based on osmosis, b.p. and alteration of v.p. are unsuitable. A viscometric method is discussed in detail and illustrated by an experimental investigation of the solutions of a number of samples of nitrocellulose (from cotton, cellulose and artificial silk) in butyl acetate, rubber in toluene and polyacrylonitrile in dimethylformamide. The $\eta_{spec.}$ of the solutions are measured over ranges of temp. and concn. using an Umstätter free-flow viscometer.
H. L. WHITEHEAD

694. **Water-soluble acids of the latex of *Hevea brasiliensis*.** E. M. Bevilacqua (*Rubb. World*, 1954, **131** [1], 69–70, 75).—The analysis of latexes of *Hevea brasiliensis* for the acidic constituents is described. Ammonia is removed from the serum (to 0·1 per cent.) at 90° C, and the pH is adjusted to 4·0 by the addition of a strong acid ion-exchange resin. The filtered serum is passed through a column of the same resin and then through a column of an ion-exchange resin in the hydroxyl form; the passage through the columns is repeated to give a product of pH 8·9. The acids are recovered from the anion-exchange resin by elution with aq. NH_3 soln. A solution of the ammoniun salts containing 200 milli-equiv. of acid is passed slowly through a column of a strong base ion-exchange resin, washed with water and then by a series of solutions of ammonium carbonate of increasing concentration; monobasic acids are completely removed by a 0·05 per cent. solution, dibasic by a 1·5 per cent. and tribasic by a 3 per cent. The eluate is collected in 100-ml fractions, which are titrated after removal of CO_2 and excess of NH_3. The component acids are identified from the titration curves in the usual way. Malic and citric acids were found in a 1-yr. old latex; malic and succinic acids in a 20-day old latex; pyrrolidonecarboxylic acid is also present. J. L. PROSSER

See also Abstracts 544, 598.

4.—BIOCHEMISTRY
INCLUDING DRUGS, FOOD, SANITATION, AGRICULTURE

Blood, Bile, Urine, etc.

695. **A new method for detecting and measuring carbon monoxide in air, blood and lungs.** V. Tomberg (*Experientia*, 1954, **10** [9], 388–389).—The method has a sensitivity of 0·01 p.p.m. for CO in air. Mercuric oxide is heated in an oven reaction chamber, where it reacts with CO and releases proportional amounts of mercury vapour, which are measured quantitatively in a u.v. electronic spectrophotometer. The reaction chamber and the u.v. meter are assembled in a portable unit. The amount of CO in blood is measured by decomposing a sample of blood in a calibrated flask with H_2SO_4 and drawing the gas mixture through the unit; the CO in alveolar air is measured by means of breathing bags. R. S. TONKS

696. **Direct determination of calcium in serum and urine with the aid of the photo-electric colorimeter.** N. J. Poulie (*Chem. Weekbl.*, 1954, **50** [41], 698–701).—A sample of 0·5 ml of serum, diluted with 5 ml of water and treated with 0·2 ml of *N* NaOH and 0·5 ml of 0·2 per cent. murexide solution, is titrated with a 0·001 *M* solution of sodium ethylenediaminetetra-acetate, added from a micro-burette. During the titration the extinction is followed at 600 mμ, and the end-point is reached when this value attains a maximum. A blank is carried out with water. The titration may also be carried out directly with urine, if the concn. of PO_4''' does not exceed 2×10^{-5} *M*. The urine is acidified with HCl and filtered. A series of dilutions from 1:100 to 1:2·5 are prepared and to 10 ml of each is added 0·4 ml of *N* NaOH, followed by 1 ml of murexide and 2 ml of sodium ethylenediaminetetra-acetate. For the actual titration, that dilution is chosen which still shows a violet colour. The titration is carried out as before, but in the initial stages must be done slowly to allow time for equilibrium to be established. G. MIDDLETON

697. **Polarographic determination of lead in blood.** A.-L. Nylander and C.-E. Holmquist (*Arch. Ind. Hyg.*, 1954, **10** [3], 183–191).—A specific and sensitive method for determining Pb in blood is described. *Procedure*—Samples of blood are evaporated to near dryness 3 times with 5 ml of conc. HNO_3, and the residue is evaporated 15 times with 1-ml portions of 30 per cent. H_2O_2 soln. Water (4 to 5 ml) and conc. HNO_3 (1 ml) are added, and

then 5 ml of ammonium citrate soln. are added while heating. The solution is neutralised and a solution of ammonium citrate and KCN is added; Pb is then extracted with dithizone in CCl_4 (a fresh solution containing 4 to 6 mg per litre). The Pb - dithizone complex is decomposed and the Pb extracted with 5 ml of *N* HCl; the acid is placed in an electrolytic vessel and heated at 90° C for 5 min. to remove CCl_4, which interferes with the polarography. Oxygen is removed by passing N_2 for $\frac{1}{4}$ 30 min.; polarography is carried out over the range −0·25 to −0·7 V with a drop time of 4·2 sec. A calibration curve is made for blood samples to which known amounts of Pb have been added.

C. E. SEARLE

698. **Indirect method for determination of serum inorganic sulphate by flame spectrophotometry.** R. D. Strickland and C. M. Maloney (*Amer. J. Clin. Path.*, 1954, **24** [9], 1100–1102).—The sulphate is pptd. from an aliquot of protein-free filtrate, as the Ba salt, by the addition of a known amount of $BaCl_2$. The pptd. $BaSO_4$ is removed by filtration and the residual Ba in the sample is determined by flame spectrophotometry. The amount of SO_4'' is calculated from the decrease in Ba concn. The use of uranyl acetate reagent for preparing the filtrate has the additional function of pptg. the inorganic phosphate, which would otherwise interfere.

R. S. TONKS

699. **Conductimetric titration method for determination of serum chloride.** H. A. Teloh (*Amer. J. Clin. Path.*, 1954, **24** [9], 1095–1099).—Serum protein is precipitated with absolute ethanol and removed by centrifugation. The supernatant ethanol diluted with water is titrated with standard $AgNO_3$. The end-point of the titration is determined by the increase in conductance, caused by the presence of excess of $AgNO_3$. Normal levels of serum chloride by this method range from 97·4 to 106 milli-equiv. per litre (mean, 101·0 milli-equiv. per litre; standard deviation, ± 2·36).

R. S. TONKS

700. **A simple method for the estimation of inorganic blood-iodine.** E. Klein (*Biochem. Z.*, 1954, **326** [1], 9–13).—Previous estimations of inorganic iodide in serum have been expressed as μg of iodine per cent. calculated as the difference between total serum I and protein-bound I. For most investigations of iodine metabolism, it is necessary to use a more direct method and to avoid errors due to the presence of di-iodotyrosine, which is not included in the protein-bound I estimation. The method proposed is based on the iodide catalysis of the reduction of ceric sulphate by arsenous acid. *Procedure*—Serum (2 ml), H_2O (10 ml) and 68 per cent. trichloroacetic acid (1 ml) are mixed, allowed to stand for 10 min. and then centrifuged. The supernatant liquid is filtered into a 20-ml graduated tube and the sediment is washed with H_2O (8 ml) containing 68 per cent. trichloroacetic acid (0·8 ml), the washings are transferred to the tube and the volume made up to 20 ml with H_2O. Two 5-ml portions of the solution are taken and one is treated with 0·1 ml of KI solution (0·1 ml = 0·025 μg of I) and the other with 0·1 ml of H_2O. To each is added arsenous acid reagent (0·4 ml) [As_2O_3 (3·71 g) dissolved in *N* NaOH (50 ml), H_2O (200 ml) added and 70 per cent. H_2SO_4 till neutral; then 70 per cent. H_2SO_4 (50 ml) is added and the soln. made up to 500 ml with H_2O]. The mixtures are heated to 37°C and ceric sulphate solution is added (0·5 ml) [ceric sulphate (12 g) in 3·5 *N* H_2SO_4 (500 ml)]. After 10 min., the colours are determined in a photometer. The amount of iodide in each test solution (expressed as μg of I) is determined by reference to a calibration curve and the difference between the two values is then expressed as μg of I per 100 ml of serum. The influence of salts, trichloroacetic acid and Na_2SO_4 on the catalysis is reported.

G. W. CAMBRIDGE

701. **Interference by amino-acids in the estimation of sugars by reductimetric methods.** R. E. Strange, F. A. Dark and A. G. Ness (*Biochem. J.*, 1955, **59** [1], 172–175).—Interference by amino-acids in the reductimetric determination of glucose is investigated; alkaline Cu and $Fe(CN)_6'''$ reagents are used. The Cu reagent is appreciably reduced by cystine, tyrosine and tryptophan; amino-acids usually affect the Cu reagent less than the $Fe(CN)_6'''$ reagent. Casein hydrolysate alone has little effect on the Cu reagent, but in the presence of glucose it has a reducing action. Precipitation by Ba^{++}, Cd^{++} or Hg^{++} does not substantially decrease the reducing power of a solution of acid-hydrolysed casein using the method of Fujita *et al.* (*Brit. Abstr. A*, 1932, 75). A method is described in which a synthetic cation-exchange resin (Amberlite IR–120) is used, allowing accurate determination of glucose in the presence of large amounts of amino-acids.

J. N. ASHLEY

702. **The quantitative determination of fructose with skatole and hydrochloric acid.** B. M. Pogell (*J. Biol. Chem.*, 1954, **211** [1], 143–147).—A method is described for the determination of fructose; it is based on the purple colour given by this sugar with skatole and aq. HCl. *Procedure*—The sample is mixed with 0·04 per cent. skatole in 95 per cent. ethanol, and ice-cold 30 per cent. aq. HCl is then added. The mixture is heated at 37° C for 40 min.; the colour intensity is then determined spectrophotometrically against a water control at 510 mμ. The amount of fructose is calc. from the optical density of a standard fructose solution. Other sugars do not react significantly at the low temp. used. The method has been applied to mouse-liver homogenate, oxalated human blood, human sera, urine and mixtures of fructose and glucose.

J. N. ASHLEY

703. **A colorimetric method for the estimation of acetoacetate.** P. G. Walker (*Biochem. J.*, 1954, **58** [4], 699–704).—A colorimetric method for the determination of 1 to 10 μg of acetoacetate is described. It is based on the reaction between acetoacetic acid and diazotised *p*-nitroaniline, when NN′-bis-(*p*-nitrophenyl)-C-acetylformazan is formed; under the specified conditions this reaction is reproducible and quant. The formazan is extracted with ethyl acetate and the optical density of the extract is determined spectrophotometrically at 440 mμ; the amount of acetoacetate is then ascertained from a standard graph (which is linear for 1 to 20 μg per ml). Acetone, ethanol, glucose, soluble barbitone, 2:4-dinitrophenol, β-hydroxybutyric acid, α-oxoglutaric acid, pyruvic acid and dehydroascorbic acid, do not interfere. Ascorbic acid interferes, but is eliminated by preliminary oxidation with air in the presence of cupric acetate.

J. N. ASHLEY

704. **Note on the chromatographic identification of blood keto-acids in animals poisoned with arsenite and alloxan.** M. F. S. El-Hawary and R. H. S. Thompson (*Biochem. J.*, 1954, **58** [4], 518–520).—When the keto-acids, pyruvic and acetoacetic acids, in blood are determined by paper-chromatographic

methods using the 2:4-dinitrophenylhydrazones of these acids, three spots are obtained. The two more rapidly moving spots are due to pyruvic acid 2:4-dinitrophenylhydrazone; a safe estimate of the pyruvate content is obtained by summation of the values obtained from both spots. High concn. of acetoacetate are unlikely to interfere in the chromatographic determination of blood pyruvate.
J. N. ASHLEY

705. **Studies on the volatile fatty acids of sheep blood with special reference to formic acid. [Determination of volatile fatty acids.]** E. F. Annison (*Biochem. J.*, 1954, **58** [4], 670–680).—A method is described for the complete analysis of the volatile fatty acids in 10 to 20 ml of blood. The sample is laked with water and deproteinised with HPO_3. The filtrate is neutralised with KOH and, after adjusting to pH 2·8 to 3·0, the mixture is distilled in a Markham still. The distillate is neutralised (phenolphthalein) with 0·01 *N* NaOH and then evaporated to give the Na salts. These are mixed with aq. H_3PO_4, and the mixture is transferred to gas - liquid partition columns, for the determination of each acid by chromatographic analysis.
J. N. ASHLEY

706. **A method for the estimation of glycerides in blood.** R. D. Stewart (*Canad. J. Biochem. Physiol.*, 1954, **32** [6], 679–683).—A fairly rapid method is described for the determination of the neutral glycerides in 0·1 ml of blood. The lipids are extracted with ethanol - ether, dissolved in $CHCl_3$ and hydrolysed. The liberated glycerol is then oxidised with HIO_4 and the resulting formaldehyde is distilled into aq. Na_2SO_3. The distillate is treated with chromotropic acid and the resulting colour is measured at 540 mμ on a photo-electric colorimeter; the amount of lipid is ascertained from a standard graph. A correction for formaldehyde derived from phospholipid may be applied, if the blood-lipid phosphorus level is known. J. N. ASHLEY

707. **Electrophoresis of serum and urine proteins on filter-paper strips and agar jelly with the bridge unit.** E. Kawerau (*Analyst*, 1954, **79**, 681–688).—The bridge unit for paper chromatography previously described (Kawerau, *Brit. Abstr. C*, 1951, 237) has been adapted for electrophoresis; the technique for electrophoresis of serum proteins on filter-paper has been investigated. Results show that even at high voltages the temp. rise is negligible and that the resolving power depends mainly upon the cellulose - protein ratio and the density of the paper. The uses of the bridge for serum on filter-paper and on agar jelly and urine on filter-paper are described.
A. O. JONES

708. **Tests on the quantitative determination of serum protein fractions in paper electrophoresis by dye elution.** F. J. Keuning (*Chem. Weekbl.*, 1954, **50** [41], 702–704).—The strips are dried at 110° C for 10 min. and fixed for 30 min. in a mixture of formaldehyde solution (50 ml), glacial acetic acid (50 ml) and 96 per cent. ethanol (400 ml). After a brief rinse in 0·25 per cent. acetic acid, the staining is done for 60 min. in an aq. solution containing 0·05 per cent. of bromophenol blue, 1 per cent. of $HgCl_2$ and 2 per cent. of glacial acetic acid. After being washed 3 to 5 times in 0·25 per cent. acetic acid (5 to 10 min.), the strips are dried and, when nearly dry, are blued in ammonia vapour. The colour taken up by albumin is 1·6 times greater than that by γ-globulin, and a corresponding factor must be allowed. Results on known mixtures show satisfactory agreement. G. MIDDLETON

709. **Reproducibility of the paper-electrophoretic protein determination.** M. J. Schulte (*Chem. Weekbl.*, 1954, **50** [41], 704).—Reproducibility of protein determinations (800) by paper electrophoresis is good. The maximum range, of not more than 10 per cent., occurred with the lowest values (α_1 and α_2-globulins); with albumin, the range was $\pm$ 1 to 2 per cent. Indirect evidence for the reliability of the method was obtained from the condition of the patients. G. MIDDLETON

710. **The colour-binding power of albumin and γ-globulin for azocarmine B.** J. van Gool (*Chem. Weekbl.*, 1950, **50** [41], 704–707).—A correction factor of from 1·0 to 1·8 has been given for the greater absorption of azocarmine B by albumin as compared with γ-globulin, but experiments (27 tests in duplicate) showed a range of 1·35 to 1·64, with a mean value of 1·48. Since 1·7 per cent. of the extinction of the albumin is calculated as γ-globulin, this factor should be increased to 1·55, and a mean value of 1·5 is recommended. There is a linear relation between the quantity of protein and the extinction, up to about 0·9 mg. By using the correction factor of 1·5, the albumin fraction of Howe corresponds approximately to the sum of the albumin and α_1 and α_2-globulin fractions, and the globulin fraction of Howe corresponds to the sum of the β and γ-globulin fractions of the electrophoresis.
G. MIDDLETON

711. **Graphical recording and evaluation of serum-protein analyses, with Antweiler's micro-electrophoresis apparatus.** C. Schumacher (*Chem.-Ing.-Tech.*, 1954, **26** [5], 279–281).—Antweiler's micro-electrophoretic method of separating proteins in serum is speeded up by the automatic recording of the electrophoresis curves, and by using a second device to evaluate these curves by automatic differentiation and estimation of the individual proportions. R. C. MURRAY

712. **Reproducibility of paper-electrophoretic determination of blood proteins.** M. J. Schulte (*Pharm. Weekbl.*, 1954, **89** [47–48], 849–852).—The paper-electrophoretic method for the quant. determination of serum proteins was found to give reliable results, reproducible even after prolonged periods. The results are tabulated. A. STORFER

713. **The estimation of serum lipoproteins: a micro-method based on zone electrophoresis and cholesterol estimations.** G. S. Boyd (*Biochem. J.*, 1954, **58** [4], 680–685).—A micro-method, which requires only 0·1 to 0·2 ml of serum, for the separation and determination of serum lipoproteins is described. It is relatively simple, cheap and gives reproducible results. It involves filter-paper zone electrophoresis of serum under specified conditions, which allow the two main lipoprotein fractions to be separated without separation of the "α" lipoprotein into its main "α_1" and lesser "α_2" fractions. The paper is then cut, and the concn. of each lipoprotein is assessed from the amount of cholesterol associated with it. The cholesterol is eluted, and the amount is determined by estimation of the optical density, absorptiometrically, of the coloured solution given by the sterol in acetic acid with H_2SO_4. J. N. ASHLEY

714. **A simple method for the quantitative determination of alpha and beta lipoproteins in serum by paper electrophoresis.** S. P. Gottfried, R. H. Pope, N. H. Friedman and S. DiMauro (*J. Lab. Clin. Med.*,

1954, **44** [4], 651–654).—In the procedure described, 0·01-ml and 0·04-ml amounts of serum are simultaneously submitted to electrophoresis on filter-paper strips moistened with barbiturate buffer (pH 8·55 and ionic strength 0·125), at a current of 4 mA for 24 hr. The strips are dried and then the protein and the lipoprotein patterns from the 0·01-ml and 0·04-ml amounts, respectively, are visualised by the two staining techniques described. Optical density, measured by a densitometer, is plotted against distance migrated for both patterns; the peaks in the density curve for the proteins are used to fix the position of the lipoprotein density peaks. Quantitative estimations of α and β-lipoproteins are made from planimeter measurements of the areas under the respective density peaks. W. H. C. SHAW

715. **Fat-staining of the serum lipoproteins on filter-paper.** H. Ott and E. Roth (*Klin. Wochschr.*, 1954, **32** [45–46], 1099–1100).—A method is proposed for the detection of lipoproteins in serum. Serum (0·05 ml) is subjected to paper-electrophoresis for 13 hr., and the paper is dried for 10 min. at 100° C and immersed in a deep staining tank maintained at 65° to 70° C. The staining solution contains Sudan black B (0·35 g), Blue BZL Ciba (0·35 g) in 75 per cent. ethanol (75 ml) and glycerol (25 ml), the mixture being boiled and then heated again after the addition of 4 per cent. NaOH solution (8·5 ml) and then filtered. After staining for 25 min., the strips are treated with 50 per cent. ethanol until the paper is only pale blue in colour. The density of the spots is determined with an electrophotometer in the usual way.

G. W. CAMBRIDGE

716. **Studies on lipid anticoagulants. I. Assays *in vitro*.** C. W. Mushett, D. P. J. Goldsmith and K. L. Kelley (*J. Biol. Chem.*, 1954, **211** [1], 163–168).—*In vitro* assays are described for lipid-type anticoagulants; they depend on the determination of the clotting time of a mixture of the anticoagulant prep. with 0·025 *M* $CaCl_2$, thromboplastin and plasma at 37·5° C. Two types of assay, emulsion and solution, are used. Emulsions of the anticoagulants are prepared with 0·85 per cent. aq. NaCl; solutions are prepared in the same solvent, then adjusted to pH 8·5 with NaOH. The assays are affected by the types of surfaces with which the plasma comes into contact, variations in the plasma and thromboplastin and the state of dispersion of the lipid sample. The precise assay of emulsified samples is difficult, but reproducible results are obtained with soluble samples under standardised conditions. J. N. ASHLEY

717. **Studies on uric acid and related compounds. I. Quantitative determination of uric acid in biological fluids.** F. Bergmann and S. Dikstein (*J. Biol. Chem.*, 1954, **211** [1], 149–153).—A method is described for the determination of uric acid in urine or plasma, after deproteinisation, if necessary, with $HClO_4$. Uric acid is determined, even in the presence of xanthine and substituted uric acids, by pptn. as mercuric urate in the presence of mercuric acetate and 1 per cent. acetic acid; the urate is redissolved in aq. NaCl. The optical density of the solution is determined at 290 mμ; it is a linear function of concn. of uric acid. J. N. ASHLEY

718. **The determination of urinary oestrogens by fluorescence.** J. S. McAnally, E. R. Hausman and C. Gables (*J. Lab. Clin. Med.*, 1954, **44** [4], 647–650).—An ethanolic solution of the urinary oestrogens, separated and purified by the detailed procedure described, is heated with H_2SO_4; the fluorescence developed is measured at 480 mμ (fluorescence max.) on a modified Model DU Beckman spectrophotometer, exciting radiation of wavelength 455 mμ being used. An internal oestrone standard is run with each determination. The fluorescence in both sample and standard plus sample is then destroyed by the addition of saturated aq. KNO_3 to the H_2SO_4 soln., and the residual non-oestrogen fluorescence is measured. The oestrogen content of the sample is determined from the difference in readings before and after KNO_3 treatment, by reference to the corresponding readings for standard plus sample. The applications and limitations of the method are discussed.

W. H. C. SHAW

719. **An improved method of gradient elution chromatography and its application to the separation of urinary ketosteroids.** T. K. Lakshmanan and S. Lieberman (*Arch. Biochem. Biophys.*, 1954, **53** [1], 258–281).—Details are given of an improved method of gradient elution analysis, in which the eluant is made more polar at a gradually increasing rate during chromatography so that a concave concentration gradient is obtained. Resolution is improved and "tailing" lessened by the proposed technique, which is investigated practically and theoretically. Factors influencing the efficiency of separation are discussed in detail. The method is of particular value in the chromatographic analysis of urinary ketosteroids on activated alumina, with benzene - ethanol mixtures for elution. Full practical details are given for this application of the method and the different types of chromatograms obtained are illustrated and discussed.

W. H. C. SHAW

720. **Polarography of steroids. II. Reaction of ketosteroids with hydrazines and primary amines.** M. Březina, V. Volková and J. Volke (*Chem. Listy*, 1954, **48** [2], 194–202).—The polarographic behaviour of the reaction products of dehydro*iso*androsterone, methyltestosterone and progesterone with Girard's reagent D (*cf. Brit. Abstr. C*, 1951, 409) (**I**) was studied. The buffered aq. alcoholic solutions of the dimethylglycylhydrazones formed show the presence of two forms of reducible compounds, the ratio of which depends on pH. The reduction at the dropping-mercury electrode requires 4 electrons and 4 protons. The following procedure for the determination of 17-ketosteroids is recommended. Evaporate the sample containing $\simeq$ 1 mg of the steroid in ethanol or other low-boiling solvent to dryness, treat the residue with a 10 per cent. soln. of **I** in glacial acetic acid (at least 0·1 ml per 1 mg of ketosteroid), heat for 3 min. of a steam-bath, cool, and add ethanol and, in order to adjust pH to 4·7, 0·25 *N* NaOH (3·2 ml per 0·1 ml of soln. **I**). Dilute with water to 5 ml and polarograph at $-$ 1·2 to $-$ 1·3 V *vs.* the S.C.E. To determine 17-ketosteroids in urine, heat the urine (10 ml) with conc. HCl (3 ml) at 80° C during 10 min., isolate the hydrolysis products by extracting with freshly distilled ether (2 $\times$ 20 ml), wash the extract with 2 *N* NaOH (40 ml) and water (2 $\times$ 40 ml), evaporate to dryness and proceed as above. G. GLASER

721. **Investigations of cortisone metabolism in adrenalectomised and gonadectomised women.** H. Staudinger, M. Taugner and W. Weiss (*Klin. Wochschr.*, 1954, **32** [45–46], 1082–1086).—17-Ketosteroids in urine were determined by a microchromatographic method (Dingemanse *et al.*, *J.*

Clin. Endocrin., 1946, **6**, 535 and *J. Clin. Endocrin. Metabol.*, 1952, **12**, 66; Pond, *Lancet*, 1951, **ii**, 906). A modification was introduced in which the urine was hydrolysed by heating with 10 per cent. HCl for 15 min. or by an enzymic method with β-glucuronidase. After a short period of heating, the urine was cooled and treated with 20,000 units of β-glucuronidase per 100 ml, brought to pH 4·4 to 4·6 and maintained at 37° C for 5 days. The extraction and separations were carried out as in the original method and a correction was made for unspecific coloured substances. Graphs are given to show the separation of various 17-ketosteroids from mixtures and from normal and pathological urine. G. W. CAMBRIDGE

722. **Quantitative determination of corticosteroids in urine.** Y. Aizawa (*Jikei. Med. J.*, 1954, **1** [1], 59–65).—Acidify 10 to 50 ml of urine to pH 1·2 with conc. HCl and extract 4 times with one-tenth the vol. of purified $CHCl_3$. Centrifuge the bulked chloroform extracts, adding anhydrous Na_2SO_4, if necessary, to break the emulsion, separate the $CHCl_3$ extract and wash it 3 times with one-tenth the vol. of 0·1 *N* NaOH, followed by 3 washes with the same amount of water. Dry the $CHCl_3$ extract over anhydrous Na_2SO_4 and distil under reduced pressure at $< 50°$C. Dissolve the residue in 1·0 to 5·0 ml of 50 per cent. ethanol; to a 0·5-ml aliquot in a Folin-Wu sugar tube, add 0·5 ml of water, 1 ml of $K_3Fe(CN)_6$ soln. (0·1 per cent.) and 1 ml of Na_2CO_3 soln. (1 per cent.). Heat in a bath of boiling water for 10 min. and cool under a tap. Add 1 ml of $FeCl_3$ soln. (0·1 per cent. in 3 per cent. acetic acid) and set aside for 15 min. Make the vol. up to 10 to 25 ml and measure the colour at 650 to 660 mμ. Recovery of deoxycorticosterone acetate from urine and water averaged 97 per cent. The method is claimed to be more sensitive and requires less urine than existing methods. H. F. W. KIRKPATRICK

723. **Symptomatic uroporphyrinuria.** T. K. With and H. C. A. Petersen (*Lancet*, 1954, **ii**, 1148–1151).—The urine of 250 patients was examined for coproporphyrin and total porphyrin contents, the difference being regarded as non-coproporphyrin porphyrins (N.C.P.). The methods used were Askevold's modification (*Scand. J. Clin. Lab. Invest.*, 1951, **3**, 318) of the method of Sveinsson, Rimington and Barnes (*Ibid.*, 1949, **1**, 2) for total porphyrin and coproporphyrin. The method of determining porphobilinogen by measurement of the total porphyrin before and after boiling the urine, whereby the porphobilinogen is transformed into uroporphyrin III, was found to be unreliable, owing to destruction of porphyrin. Paper-chromatographic separation of the porphyrins was effected with lutidine by the technique of Nicholas and Rimington (*Ibid.*, 1949, **1**, 12). P. HAAS

724. **Simultaneous determination of kynurenine and *p*-phenetidine in human urine.** M. Špaček (*Canad. J. Biochem. Physiol.*, 1954, **32** [6], 604–609).—A simple sensitive fairly specific and accurate method for the simultaneous determination of kynurenine and *p*-phenetidine in human urine is described. The kynurenine is converted, by boiling with aq. NaOH soln., into *o*-aminoacetophenone, which is determined spectrophotometrically; simultaneously, *p*-phenetidine is liberated from its conjugate, and determined in the same way. *Procedure*—Add NaOH (10 to 15 g) to the urine (100 ml; if high absorption is expected, use 25 or 50 ml and dilute to 100 ml) and distil. Extract 50 ml of the distillate with ether (15 ml) and determine the extinction (optical density, *k*) at 296, 308, 320, and 360 mμ. The following formulae are then used: $k_{360m\mu} \times 0{\cdot}59 =$ mg of kynurenine per 100 ml of urine, and $1{\cdot}35 \times (2k_{308m\mu} - k_{296m\mu} - k_{320m\mu})$ gives mg of *p*-phenetidine per 100 ml. J. N. ASHLEY

725. **The determination of N-methyl-2-pyridone-5-carboxyamide in human urine.** J. M. Price (*J. Biol. Chem.*, 1954, **211** [1], 117–124).—A rapid method for the determination of N-methyl-2-pyridone-5-carboxyamide in urine from patients with glycosuria, nephritis, uraemia, pyelonephritis, severe haematuria and neoplastic diseases is described. The urine is passed through a column of alternate layers of Dowex 1 (in hydroxide form) and Dowex 50 (in the acid form); this removes practically all of the u.v. light-absorbing materials except the pyridone, and this is determined spectrophotometrically in the effluent against water controls at 258, 295 and 310 mμ. When much blood or other protein is present, this is removed first by adding solid trichloroacetic acid and filtering. Related metabolites and several chemically related compounds do not interfere, except the isomeric N-methyl-2-pyridone-3-carboxyamide which has not been found in human urine. Sixteen determinations can be made in a day. J. N. ASHLEY

726. **Paper-chromatographic detection of small amounts of barbiturates.** C. Riebeling and H. Burmeister (*Klin. Wochschr.*, 1954, **32** [43–44], 1057–1058).—A method is described for the extraction of barbiturates from urine and their identification on a chromatogram by means of their fluorescence in u.v. light. Urine (10 to 15 ml, or more if dilute) is brought to pH 11 with 0·5 *N* NaOH. It is then shaken three times with ether (10 ml). Barbiturates may concentrate in the turbid boundary layer, which must be worked up with the urine. The urine is acidified with *N* HCl to pH 3 and shaken twice with ether (10 ml). The ether is centrifuged and evaporated in a porcelain dish. The residue is taken up in 0·05 *N* NaOH (0·05 ml) and transferred to filter-paper in two portions, 0·01 and 0·04 ml. Control spots of a 0·1 per cent. soln. of barbitone (50 μg) are also placed on the paper. The chromatograms are run for 19 hr. in butanol - 25 per cent. ammonia - water (84:8:8). The paper is dried, sprayed with 0·5 *N* NaOH and examined under u.v. light.

The R_F values are 1·0 for barbitone and barbitone sodium, 1·08 to 1·14 for phenobarbitone, 1·28 to 1·34 for cyclobarbitone and 1·21 to 1·24 for bromopropenyl*iso*propylbarbituric acid. The limit of detection is 15 to 20 μg. G. W. CAMBRIDGE

727. **Micro-method for the quantitative determination of plasma prothrombin and proconvertin.** H. Dyggve and E. Lund (*Acta Med. Scand.*, 1954, **150** [3], 208–214).—A method has been devised for the micro-estimation of plasma prothrombin and proconvertin in 0·1 ml of blood. The usual methods require 4 to 5 ml of blood and are not convenient for estimations in infants, or when frequent determinations are required. The method is based on the macro-method of Owren and Aas (*Scand. J. Clin. Lab. Invest.*, 1951, **3**, 201); comparisons are given of the results obtained by both methods. The influence of the haematocrit value of the blood on the final concentration of Na citrate in the test plasma is less in the micro than in the macro-method. *Procedure*—Capillary blood (0·1 ml) is

taken into a pipette containing Na citrate solution (A); the mixture is rapidly transferred to a small tube and centrifuged at 2000 r.p.m. for 5 min. The supernatant-plasma dilution is used for testing and 0·2 ml is mixed with thromboplastin (0·2 ml) (Owren, *Scand. J. Clin. Lab. Invest.*, 1949, **1**, 81) and prothrombin-free and proconvertin-free ox plasma (0·2 ml) (Owren and Aas, *Scand. J. Clin. Lab. Invest.*, 1951, **3**, 201), the mixture is heated at 37° C for 5 min. and $CaCl_2$ solution (0·2 ml) is added. The $CaCl_2$ solution may be 20 to 40 m*M* and a suitable concentration is chosen to give the shortest coagulation time. The time for clotting is determined in the usual way and the percentage of thrombin-proconvertin is estimated by means of a correlation graph. This is determined by using 1, 5, 10, 12·5, 25 and 50 per cent. dilutions of normal plasma in the above test, and plotting the dilutions on the abscissa and clotting time in seconds as the ordinate on log - log paper. A new graph must be plotted whenever new ox plasma or thromboplastin is used. When specific determination of prothrombin is required, a proconvertin solution (Owren and Aas, *loc. cit.*) (0·4 ml) is used in place of the ox plasma and the test is carried out immediately after addition of all the reagents. For the specific determination of proconvertin, the following technique is applied. Plasma (0·1 ml) is treated with sodium citrate solution (B) (0·1 ml), proconvertin-free ox plasma (0·2 ml), thromboplastin (0·2 ml) and $CaCl_2$ solution (0·2 ml); the time for clotting is determined as before. A correlation graph is derived from plasma dilutions as given above, the activity of 0·2 ml of test plasma plus 0·2 ml of soln. B being taken as 100 per cent. in this test.

The citrate solutions have the following composition—

A = 34·2 m*M* Na citrate solution (231 ml) + Owren's buffer (200 ml) + 0·9 per cent. NaCl solution (569 ml), and

B = 34·2 m*M* Na citrate solution (192 ml) + Owren's buffer (200 ml) + 0·9 per cent. NaCl (608 ml).

Owren's buffer = Na diethylbarbiturate (11·75 g), NaCl (14·67 g), 0·1 *N* HCl (430 ml) and distilled H_2O (1570 ml). G. W. CAMBRIDGE

728. **Studies on serum enzymes in diseases of the liver. (The activity of phosphohexose isomerase, aldolase and alkaline phosphatase.)** F. H. Bruns and W. Jacob (*Klin. Wochschr.*, 1954, **32** [43–44], 1041–1044).—The Bruns and Hinsberg method for the estimation of serum phosphohexose isomerase (**I**) (*Anal. Abstr.*, 1955, **2**, 161) has been applied to the study of enzyme changes in liver disorders. The amount of fructose 6-phosphate (**II**) formed from glucose 6-phosphate (**III**) by serum under standard conditions is estimated photometrically in the trichloroacetic acid filtrate by the method of Roe (*J. Biol. Chem.*, 1934, **107**, 15). *Procedure*—Serum (10 ml) is incubated for ½ or 1 hour at 37° C with barbitone buffer at pH 7·8 (1 ml) and 0·06 *M* soln. of **III** at pH 7·8 (1 ml). Proteins are then pptd. by the addition of 7 per cent. trichloroacetic acid (7 ml) and the mixture is filtered. A blank determination is made by adding the substrate after deproteinisation. To 2 ml of the filtrate (≡ 0·2 ml of serum), 0·1 per cent. resorcinol in pure ethanol (2 ml) is added and 30 per cent. HCl (6 ml). After mixing, the soln. is heated for 8 min. at 80° C in a water-bath and then cooled for 5 min. in water. A red colour develops and the extinction is measured at 490 mμ with a spectrophotometer. When the **I** content is high, greater dilution of serum should be used, as the calibration curve is not linear over a wide range. The calibration curve is constructed by use of **II**, and the **I** activity is expressed as the amount of **II** formed by 1 ml of serum in 1 hr. at 37°C (1μ-mole of **II** = 260 μg = 22·4 μl). Serum can be stored at 3° to 6° C for 3 to 4 days or at −15° C for 3 to 4 weeks without loss of activity. G. W. CAMBRIDGE

729. **Simple determination of the pseudocholinesterase activity in blood serum.** C. L. Harders and J. M. van Mulken (*Pharm. Weekbl.*, 1954, **89** [47–48], 845–848).—The method described, for the determination of pseudocholinesterase activity in the blood serum of normal persons, is based on the measurement of acetylcholine liberated from the serum. The pseudocholinesterase activity is expressed as the amount of 0·01 *N* NaOH (in ml) required to neutralise the acid liberated from acetylcholine in 1 ml of serum per min. The results obtained by various methods are not directly comparable, the mean values varying within 20 per cent. A. STORFER

730. **The determination of ^{65}Zn in tissues with Geiger and scintillation counters.** T. E. Banks, R. Tupper, R. W. E. Watts and A. Wormall (*Biochem. J.*, 1955, **59** [1], 149–152).—A simplified method is described for the determination of ^{65}Zn in tissues, by the use of the Veall-type liquid counter. Simple acid digests are used; the modified method also requires less manipulation and transference of solutions. A scintillation counter suitable for the determination of ^{65}Zn in liquids or tissue homogenates is described; a Tl-activated NaI crystal is used as the phosphor. When the scintillation method is used, the background count rate is much larger than with Geiger - Müller tubes, but the ratio of net count rate of sample to background count rate is larger and more favourable. Two further advantages of the modified method are that the glass dishes containing the final solution are of simple design and very easily cleaned. Separate dishes can be used for each sample. The method is of general application and gives satisfactory results. J. N. ASHLEY

731. **Some applications of the van Slyke manometric technique.** G. Ingram (*Chem. & Ind.*, 1954, [50], 1527–1531).—Applications of the van Slyke manometric technique are discussed, including determination of amino nitrogen in amino-acids and peptides. The following determinations have been carried out: 0·3 to 3·5 mg of C in organic matter, with an accuracy of ± 0·5 per cent.; CO in air, with an error of < 5 per cent., by absorption in Winkler's cuprous chloride soln.; and carbonates and CO_2. Details of the apparatus are illustrated and described. O. M. WHITTON

732. **The separation of amino-acids by buffered circular-paper chromatography.** K. Krishnamurthy and M. Swaminathan (*Current Sci.*, 1954, **23** [7], 223–224).—A preliminary account is given of the separation of commonly occurring amino-acids by buffered circular-paper chromatography. The apparatus described by Giri and Rao (*J. Indian Inst. Sci.*, 1952, **34**, 95) and the solvents and buffers described by McFarren (*Brit. Abstr. C*, 1951, 217) were used, and the chromatograms were developed twice as described by Jeanes *et al.* (*Brit. Abstr. C*, 1951, 316) for the separation of carbohydrates. After twice developing for 8 hr. and subsequent

drying for 3 min. at 80° C, the separated amino-acids were located by spraying with acetone containing 0·2 per cent. ninhydrin and 5 per cent. acetic acid, and the paper was dried at 80° C for 5 min. *m*-Cresol, saturated with buffer of pH 8·4, gave a clear separation of alanine, arginine, tyrosine, histidine, valine, methionine and phenylalanine. Phenol, saturated with buffer of pH 12·0, gave a satisfactory separation of aspartic acid, glutamic acid, serine, glycine, threonine and alanine.

S. C. JOLLY

733. **Chromatographic separation of the diastereo-isomerides of lanthionine. Determination of lanthionine.** S. Blackburn and G. R. Lee (*Chem. & Ind.*, 1954, [41], 1252).—When amino-acid mixtures, or protein hydrolysate containing lanthionine, are applied to a 100-cm × 0·9-cm column of Dowex 50 ion-exchange resin, developed with a buffer of pH 3·2, lanthionine emerges between the serine and glutamic acid fractions. The eluate is collected in 1-ml portions; lanthionine is estimated by adding to each portion 1 ml of glacial acetic acid and 1 ml of ninhydrin reagent of pH 1·0 (prepared by dissolving 2·5 g of ninhydrin in 4 ml of 6 *M* H_3PO_4 and 60 ml of glacial acetic acid). The mixture is heated on a water-bath for 10 min., diluted with 2 ml of glacial acetic acid, and the optical density of the soln. is determined; the amount of lanthionine present is read from a standard curve. Complete separation of L- and *meso*-lanthionine has been achieved by using a 300-cm × 1-cm column at room temp.

G. R. WHALLEY

734. **The characterisation of guanidine derivatives of biological origin by electrophoresis and paper chromatography (chromato-electrophoresis).** S. Lisitzky, I. Garcia and J. Roche (*Experientia*, 1954, **10** [9], 379–381).—Paper chromatography has been used in conjunction with electrophoresis in a perpendicular direction to characterise various guanidine derivatives and citrulline. Definite spots can be isolated with a satisfactory selectivity by this procedure at a higher speed than by two-dimensional chromatography. The use of coloured reactions (ninhydrin, diacetyl-1-naphthol and *p*-dimethyl-aminobenzaldehyde) on the same electrochromatogram has been successful.

R. S. TONKS

735. **The application of the ceric sulphate - arsenous acid reaction to the detection of thyroxine and related substances.** C. H. Bowden, N. F. Maclagan and J. H. Wilkinson (*Biochem. J.*, 1955, **59** [1], 93–97).—Methods are described for the detection of inorg. and org. I-containing compounds, in solution and on paper chromatograms, with the $Ce(SO_4)_2$ - As_2O_3 reagent; they are based on decolorisation of this reagent. At room temp., 0·2 μg of KI and 2·0 μg of thyroxine are detectable in solution and 0·01 μg of KI and 0·1 μg of thyroxine on paper chromatograms. Methods are described for making permanent records of the chromatograms. The general procedure can be adapted for the semi-quant. determination of 0·1 to 0·4 μg of thyroxine. The method probably depends on liberation of I from the compounds by oxidation with $Ce(SO_4)_2$. With the exception of KIO_3, only those compounds which readily give I when treated with $Ce(SO_4)_2$ can be detected on paper with this reagent. The method is relatively insensitive to aliphatic iodo compounds; the reaction is given by all the aromatic compounds examined that contain one or more I atoms ortho to an electron-donating substituent, such as a hydroxyl or amino group.

J. N. ASHLEY

736. **The thyroxine content of thyroactive iodinated proteins as determined by a radioactive-isotope dilution technique.** E. P. Reineke (*J. Dairy Sci.*, 1954, **37** [10], 1227–1232).—Details are given of an isotope dilution procedure with ^{131}I-labelled thyroxine for the determination of thryoxine in iodinated protein. It is shown that, during hydrolysis with 40 per cent. aq. $Ba(OH)_2$ and the subsequent purification and crystallisation procedures, no significant interchange of iodine takes place between radioactive thyroxine and inorganic iodine. Results, determined by the isotope dilution method on seven samples of iodinated casein, gave an average thyroxine content of 1·04 ± 0·049 per cent., compared with 3·24 ± 0·429 per cent. of apparent thyroxine indicated by the *n*-butanol extraction procedure detailed previously (*Brit. Abstr. C*, 1946, 99). The discrepancies are mainly attributed to the inclusion of di-iodothyronine and tri-iodothyronine in results obtained by the latter method. The preparation of labelled thyroxine from $Na^{131}I$ and casein is described.

W. H. C. SHAW

737. **An assay method for antidiuretic hormone based on a more specific response index.** J. D. Crawford and B. Pinkham (*Endocrinology*, 1954, **55** [5], 521–529).—A description is given of an improved method for the assay of antidiuretic hormone (ADH) in rats. The activity is determined by its effect, under controlled conditions, on the ratio of water to total osmotically effective solute in the urine as determined from freezing-point observations. A rectilinear relationship is obtained when the results, expressed as the percentage decrease in the water concentration of the urine, are plotted against the logarithm of the dose in the range 0·1 to 0·4 of a milli-unit of Pitressin. A high degree of sensitivity and precision is attainable by the method described and the increased specificity for ADH is discussed.

W. H. C. SHAW

738. **Assay of adrenocortical hormones on the thymus of the weanling rat.** N. R. Stephenson (*Canad. J. Biochem. Physiol.*, 1954, **32** [6], 689–702).—Involution of the thymus gland of intact weanling rats is used as the criterion of the response to 11-oxycorticosteroids. Other substances known to cause thymus atrophy have dosage - response relationships that differ significantly from those observed with 11-oxycorticosteroids. Thymus involution is produced more rapidly, and at lower dose levels, by adrenal corticoids than by any other steroid hormone preparation examined. Methods are described for increasing the slope of the dosage - effect line and for reducing the error term of the assay. The standard error of the potency ratio is usually $< \pm 15$ per cent., when 40 intact Wistar-strain female 21 to 23-day old rats are used in a 2-dose assay design. There is a linear relationship between log-dose and the thymus wt. per 100 g of rat. Results given by this method do not differ significantly from those obtained by the method using deposition of reducing substances in the liver of adrenalectomised male rats as the criterion of response. The advantages of the new method are that intact rats are used, and chemical analyses of the tissues are not needed.

J. N. ASHLEY

739. **Chromogenic values of 17-hydroxycorticosteroids in a modified Porter - Silber reaction.** H. Wilson and R. Fairbanks (*Arch. Biochem. Biophys.*, 1954, **53** [1], 71–76).—Details are given of a semi-micro modification of the phenylhydrazine - H_2SO_4 method for 17:21-dihydroxy-20-oxosteroids

described by Porter and Silber (*Brit. Abstr. C*, 1951, 215). Of fifteen compounds tested, cortisone, dihydrocortisone and tetrahydrocortisone were strongly chromogenic. Hydrocortisone and Reichstein's compound-S gave one-third, and the four corresponding dihydro compounds one-tenth of the colour of cortisone; two tetrahydro derivatives of the two compounds showed little reaction. *Procedure*—Measure duplicate 0·2-ml quantities of absolute ethanol (for blank) and of standard and test solutions, containing the equivalent of 4 to 20 μg of cortisone in absolute ethanol, into calibrated 10-mm × 75-mm test tubes. To one tube of each pair, add 1·8 ml of H_2SO_4 reagent (183 ml of conc. H_2SO_4 mixed with 100 ml of water) and to the other, 1·8 ml of phenylhydrazine reagent (60 mg of recryst. phenylhydrazine hydrochloride in 90 ml of the H_2SO_4 reagent). Stir each solution with a glass rod left *in situ* and allow to stand for 3 hr. in the dark. Stir again and then measure the optical-density difference for each pair at 420 mμ. Deduct the optical-density difference for the blank pair from that for both standard and test. Calibration is rectilinear for cortisone (5 to 30 μg) and for hydrocortisone (10 to 60 μg). W. H. C. SHAW

740. **Iodimetric assay of penicillinase.** C. J. Perret (*Nature*, 1954, **174**, 1012–1013).—A rapid accurate and sensitive method for the assay of penicillinase is described. It depends on the destruction of 0·005 *M* Na benzylpenicillin by the enzyme in 0·2 *M* phosphate buffer at pH 6·5 and 30° C. After a definite reaction time, a reagent, which consists of 0·0166 *N* I and 0·06 *M* KI in 1·75 *N* Na acetate buffer (pH 4·00), is added, and then the excess of I is titrated with 0·0166 *N* $Na_2S_2O_3$. One ml of 0·0166 *N* I is equivalent to 2 μ-mole of penicillin destroyed. A "unit dose" of penicillinase is that amount of enzyme which destroys 1 μ-mole of penicillin per hr. Corrections for spontaneous decay of penicillin and the I uptake of crude enzyme samples are determined from controls. By starting the assays at intervals of 3 min., two operators can carry out 10 assays in 1 hr. Before the assay, the approximate activity of the enzyme is determined by a modification of the assay method. Fifty of these determinations can be carried out by 2 workers in 1 hr. Modifications of the method are described for working over a wide range of conditions. The assay is designed for penicillinase from *B. cereus* NRRL-569; it may be applicable in principle to the enzyme from other sources. J. N. ASHLEY

See also Abstracts 742, 752, 761, 762, 764.

Drugs

741. **Chromatography of alkaloidal bases on acidic alumina.** P. Tunmann and W. Hubmann (*Arch. Pharm., Berlin*, 1954, **287** [5], 281–290).—Alumina (5 g, 70 to 90-μ granule size), is suspended in 30 ml of $CHCl_3$ with 0·02 g of methyl red at various constant temp. between 20° and 40° C and stirred and 2 litres of dry HCl is passed through. The solvent is syphoned off and the excess of indicator is washed out with $CHCl_3$. This gives a red powder which on packing (height and compression controlled) affords a column through which the passage of a definite wt. of hyoscyamine in $CHCl_3$ changes a definite length of column from red to yellow in accordance with the ion exchange expression: > Al–Cl + (BH^+) OH^- → > Al–OH + $(BH)^+Cl^-$; this result is reproducible. Statistical analyses of 32 results with hyoscyamine soln. of various strengths leads to the expression, zone length in mm = constant × wt. in mg ± mean deviation in mm. The higher the HCl - Al_2O_3 reaction temp., the shorter the column length. Determination of the titre of the column by titrating with 0·1 *N* NaOH gives an equiv. in 0·1 *N* HCl per mm. of column and from this may be calculated the theoretical zone length for a given wt. of alkaloid. For an alkaloid of p*K* value of 4·6 there is agreement with experiment, but if p*K* > 4·6 the zone length is greater and vice versa. After passage through a standard column followed by elution, estimation of alkaloid and Cl′ in the eluate leads to the following conclusions. From a mono-acid base, the eluate contains equivalent amounts of alkaloid and Cl′; a di-acid base yields more Cl′ than the mono-acid salt requires and the excess varies from alkaloid to alkaloid; the alkaloidal HCl takes longer to appear than the solubility in $CHCl_3$ predicts; less salt is obtained than alkaloid employed, suggesting adsorption. The method is possible for the acid - alkali determination of non-titratable alkaloids. F. R. MUMFORD

742. **A rapid method for the estimation of morphine.** J. M. Fujimoto, E. L. Way and C. H. Hine (*J. Lab. Clin. Med.*, 1954, **44** [4], 627–635).—In the method described, morphine is extracted from biological materials, and interfering substances are eliminated by a series of single extraction-aliquot procedures carried out under conditions that ensure the maximum specificity for morphine. The major separation of morphine is obtained by extracting with *n*-butanol from the material made strongly alkaline with KOH. The final purified extract is treated with molybdosilicic acid and aq. NH_3; the resulting colour is measured at 675 mμ. Detailed extraction procedures are given for liver and for urine, from which over-all recovery is 71 per cent. for free morphine and 68 per cent. for total morphine. At concentrations over 6 μg per ml, the error in determination is < ± 10 per cent. Morphine is identified in the purified extracts by its behaviour, when partitioned between $CHCl_3$ and aq. buffers at different pH values, and on the differences in u.v. absorption in acid and alkaline solutions. Incorrect results for morphine are obtained if the sample contains N-allylnormorphine (nalorphine), normorphine, dihydromorphinone (Dilaudid), Metopon or 4-aminosalicylic acid. W. H. C. SHAW

743. **An improvement in the gravimetric method of determination of morphine in poppy capsules.** E. Wegner (*Dtsch. ApothZtg.*, 1954, **94** [49], 1201–1202).—Lipoids extracted from the capsule may cause high results in the gravimetric determination of morphine as dinitrophenyl the ether in poppy capsules. When a correction is applied, according to the lipoid content of the extracted material, gravimetric results agree within 2 per cent. with results obtained from photometric determinations. A. R. ROGERS

744. **Determination of the purity of digitalis glycosides.** J. E. Murphy (*J. Amer. Pharm. Ass., Sci. Ed.*, 1954, **43** [11], 659–661).—The fluorimetric method of Jensen (*Brit. Abstr. C*, 1953, 24), modified by the use of propylene glycol instead of glycerol in the hydrochloric acid reagent, is used to determine steroids of the gitoxin series present as impurities in digoxin, digitoxin or lanatoside C. Two unknown glycosides respond similarly. A. R. ROGERS

745. **Noradrenaline: an identity test with nitrite.** H. Aasness (*Norges Apotheker. Tidsskr.*, 1954, **62** [6], 119–122; [7], 139–144).—The significance and chemistry of noradrenaline are discussed, and experiments in which nitrite is used for identification are described. With nitrite, a pH of 5 is most effective in detecting adrenaline in noradrenaline. The former gives a red, and the latter a yellow, colour. By using 0·10 ml of a 2 per cent. soln. of noradrenaline bitartrate with 1 ml of buffer and 0·1 ml of *M* nitrite, the colour developed after 2 min. should not be a more intense red than the colour standard (10 Co - 40 Cu) of the Dutch Pharmacopoeia (Add. I, p. 12). G. MIDDLETON

746. **The spectrophotometric estimation of total penicillins by conversion to penicillenic acid and the importance of copper in controlling the reaction.** F. G. Stock (*Analyst*, 1954, **79**, 662–670).—In the development of a modification of Herriot's method (*Brit. Abstr. C*, 1946, 268) for estimation of total penicillins, it was found that addition of Cu to the buffer soln. effected exact reproducibility. For routine estimation of solid penicillin and oral tablets containing 10,000 i.u. or more, a soln. containing 160 to 200 i.u. per ml is prepared. *Procedure*—Ten ml of the test soln. are added to 30 ml of 0·4 *M* acetic acid - sodium acetate buffer soln. containing 0·45 p.p.m. of added Cu. Six tubes, each containing 4 ml of the mixture, are immersed in boiling water for 15 min. and replicate determinations are made of the increase of optical density at 322 mμ, the increase being due to the formation of penicillenic acid. From the mean of these values and from the standard expression $E^{50\ \text{i.u. per ml}}_{1\ \text{cm}}$ at 322 mμ = 0·76, the penicillin potency is determined. The determination is repeated with acetate buffer containing no Cu and if the mean value found is lower than that found in the presence of Cu and the fiducial limits of the mean of the six values are $\simeq$ + 2 per cent. or more, then the Cu originally present in the buffer mixture can be assumed to be $\simeq$ 0·10 p.p.m. and is of no consequence. Results by this method agree with those of biological assays. A. O. JONES

747. **The assay of benzathinepenicillin by titration in a non-aqueous solvent.** C. Knight and W. H. Stephenson (*J. Pharm. Pharmacol.*, 1954, **6** [12], 1002–1004).—Benzathinepenicillin may be assayed by titration with 0·1 *N* perchloric acid in glacial acetic acid containing 2 per cent. of acetic anhydride, with crystal violet as indicator; four equivalents are required. Results are in reasonable agreement with those obtained by iodimetric, base-extraction, optical-density and microbiological determinations. A. R. ROGERS

748. **Polarographic determination of Aureomycin and Terramycin in pharmaceutical preparations.** O. Tělupilová and V. Mašínová (*Českosl. Farmac.*, 1953, **2** [7–8], 226–229; *Referativnŷĭ Zh., Khim.*, 1954, Abstr. No. 18,657).—The methods of Doskočil and Vondráček (*Chem. Listy*, 1952, **46**, 564) are applied to pharmaceutical preparations. For Aureomycin (chlortetracycline), phosphate buffer at pH 5 to 8 is the most suitable; and for Terramycin (oxytetracycline), buffer at pH 5 to 6·5. By the use of the Britton - Robinson buffer at pH 6·5, the two antibiotics can be qualitatively distinguished; the form of the reduction waves and the half-wave potentials are different. For mixtures of Aureomycin and Terramycin, the wave is similar to that of Terramycin in form, and its height corresponds to the concn. of the two substances. E. HAYES

749. **The effect of neomycin on *Brucella bronchoseptica* in the assay of polymyxin B.** N. F. Knowlden and E. Broomfield (*Antibiot. & Chemother.*, 1954, **4** [11], 1167–1173).—A statistical study is made of the effect of neomycin in the plate assay for polymyxin B with *B. bronchoseptica* (ATCC 4617) and the assay procedure described previously (*Anal. Abstr.*, 1955, **2**, 447). For materials containing both antibiotics, the neomycin content is determined in a preliminary experiment and then, by reference to the nomogram given, the result of the polymyxin-B assay is corrected accordingly. W. H. C. SHAW

750. **Analysis of barbiturates. Comparison of aqueous and non-aqueous methods.** J. C. Ryan, L. K. Yanowski and C. W. Pifer (*J. Amer. Pharm. Ass., Sci. Ed.*, 1954, **43** [11], 656–659).—For the titration of barbiturates, 0·1 *N* lithium methoxide in benzene is recommended; dimethylformamide is used as solvent and the end-point is indicated by the change in colour of thymol blue to dark blue. Sodium salts, capsules and tablets require preliminary acidification and extraction of the free acid with ether or chloroform. The method is equivalent in accuracy and precision to that of the U.S.P. XIV, but it is more rapid. A. R. ROGERS

751. **Identification of barbiturates by X-ray analysis. IX. Binary mixtures of Aethallymal and other barbiturates.** E. J. Hansen and B. Jerslev (*Dansk. Tidsskr. Farm.*, 1954, **28** [10], 220–237).—Solid soln. formation is investigated for Aethallymal (5-allyl-5-ethylbarbituric acid, m.p. 160° C) and its unstable modifications with Allypropymal (5-allyl-5-*iso*propylbarbituric acid), Diemal (barbitone), Hexemal (cyclobarbitone) and their modifications. X-ray data are given for the approx. analysis of the two first-mentioned systems; with Hexemal, mechanical mixtures are formed which allow of identification only. P. S. ARUP

752. **Contributions to the toxicological knowledge of Dromoran [levorphan], morphine, Dilaudid [dihydromorphinone], Cardiazol [leptazol], Coramine [nikethamide] and atropine with the aid of paper chromatography.** H. Kaiser and H. Jori (*Arch. Pharm., Berlin*, 1954, **287** [4], 224–242; [5], 253–258).—**I. Levorphan.** The chemical nature, qual. reactions and quant. estimation of levorphan are discussed. The hydrochloride has 2 molecules of water of crystallisation and m.p. 115° C; the free base has a m.p. of 112° to 113° C (rapid heating). A crystalline aggregate sublimate (illustrated) is obtained by long heating of the base at 115° C (Kofler block). The sublimate has a m.p. of 121° C. Micro-sublimation of the base at 130° C gives slender needles, but at 121° C both needles and aggregates appear in the sublimate (illustrated). Paper chromatography of morphine and levorphan in *n*-butanol - 0·1 *N* HCl (1:1) separates the drugs, giving spots of R_F value 0·77 and 0·31 for levorphan and morphine, respectively; the drugs are recognised by reaction with H_2O - $FeCl_3$ - $K_3Fe(CN)_6$, by their phenolic nature. Levorphan may be extracted from urine by a modified Stas - Otto process in the presence of barbiturate, and may be quant. determined colorimetrically, after treatment with *p*-nitroaniline in dil. HCl followed by $NaNO_2$ and basification. A standard curve is given for both the salt and the anhydrous base. In 31 hr., after administration of 3 mg of levorphan, 22·8 per cent.

is recovered from the urine. Isolation from urine and estimation has an accuracy of ± 10 per cent., whilst estimation in aq. soln. has an accuracy of ± 4 per cent. Levorphan in urine may also be purified for estimation by adsorption on a kieselguhr column followed by elution with $CHCl_3$.

II. Morphine and Dilaudid (dihydromorphinone). The analysis of these is discussed and photomicrographs are given of the complexes of these drugs with K - Fe (also Hg, Cu and Bi) iodides, $PdCl_2$, I_2 - KI, $PtCl_4$, $HgCl_2$ and Mayer's reagent. Paper chromatography of morphine and dihydromorphinone (as in **I**) shows little separation with R_F values of 0·31 and 0·32, respectively, with the same reagent for identification or a modified Dragendorff's reagent. Morphine and dihydromorphinone may be extracted from urine by a modified Stas - Otto process, with aq. NH_3 soln. and $NaHCO_3$, respectively, for basification. A new colorimetric estimation of morphine is detailed with *N* NaOH, 0·2 per cent. $K_3Fe(CN)_6$ and an Fe - H_3PO_4 reagent of specified composition. The accuracy is ± 8 per cent.

III. Cardiazol (leptazol) and Coramine (nikethamide). The chemical nature and toxicology of leptazol and nikethamide are discussed. Paper chromatography of these drugs using *n*-butanol - 0·1 *N* HCl (1 : 1) gives spots of R_F values 0·86 and 0·32, respectively; if no HCl is present, nikethamide has an R_F value of 0·85. The spots are recognised by a modified Dragendorff's reagent.

IV. Atropine. The paper chromatography of atropine is discussed. Chromatography of atropine gives a spot of R_F value 0·57, but after passage through the body a second spot appears with R_F 0·69. Dihydromorphinone and atropine may be separated by paper chromatography. Atropine is recognised in the chromatogram by the modified Dragendorff's reagent.

F. R. MUMFORD

753. **Ion-exchange resins in the analysis of tablets and dragees.** A. Jindra and O. Motl (*Českosl. Farmac.*, 1953, **2** [6], 190–191; *Referativnýĭ Zh., Khim.*, 1954, Abstr. No. 15,090).—The method of Bjorling (*Farm. Revy*, 1949, **48**, 281) is applied to the commercial preparations Acedicon [acetyldemethylodihydrothebaine hydrochloride], Parpanit [caramiphen], Syntropan and Trasentin. Tablets or dragees containing 20 to 100 mg of compound are treated with 50 per cent. ethanol and the resulting suspension is filtered through cotton-wool. The residue is extracted several times with 96 per cent. ethanol and the combined filtrates are passed through a column of Amberlite IRA–400 at the rate of 7 ml per min. The column is washed with 40 to 80 ml of hot ethanol and, after dilution with 40 ml of water, the eluate is titrated with 0·1 *N* HCl.

E. HAYES

754. **The analysis of ointments containing silicones.** R. Springer and R. Herzinger (*Arch. Pharm. Berlin*, 1954, **287** [4], 204–209).—The dermatological use and chemical nature of silicones are discussed and the problem of their estimation in ointments is considered. The physical characters of a number of silicones are listed and a graph is given of the variation in SiO_2 equivalent with the number of Si atoms in a silicone, enabling analysis, to give the degree of polymerisation. In the proposed analysis of a silicone-based ointment, by refluxing with trichloroethylene, filtration and washing with solvent, a filtrate is obtained containing soluble organic material and a solid residue of insoluble medicament (*e.g.*, ZnO). Volatile oil and H_2O can be determined in the usual manner. Removal of solvent gives a residue containing silicone from which fats, etc. are removed by saponification and the Si in the unsaponified residue is determined by heating to 150° C with H_2SO_4, stirring and adding HNO_3 dropwise to produce SiO_2. After dilution, removal of HNO_3 with urea and boiling, silica is removed by filtration, then dried and weighed. The weight × 1·25 gives the silicone content.

F. R. MUMFORD

755. **The application of infra-red spectroscopy to pharmaceutical analysis.** D. C. Garratt and P. G. Marshall (*J. Pharm. Pharmacol.*, 1954, **6** [12], 950–959).—A general outline of infra-red technique is given, together with examples of its value in the qualitative and quantitative analysis of pharmaceutical materials, including the determination of both theophylline and phenobarbitone in tablets of aminophylline and phenobarbitone.

A. R. ROGERS

756. **The use of oscillographic polarography in pharmacy.** J. Heyrovský (*Českosl. Farmac.*, 1953, **2** [12], 403–406; *Referativnýĭ Zh., Khim.*, 1954, Abstr. No. 22,158).—The principles of oscillographic polarography and its application to the determination of various pharmaceutical preparations are briefly reviewed.

E. HAYES

757. **Complexiметric titrations in pharmaceutical analysis. V. Determination of magnesium.** R. Přibil, J. Čihalik, J. Doležal, V. Simon and J. Zýka (*Českosl. Farmac.*, 1953, **2** [6], 184–185; *Referativnýĭ Zh., Khim.*, 1954, Abstr. No. 20,359).—Magnesium sulphate or chloride is dissolved in water and, after the addition of 5 to 8 ml of buffer soln. at pH 10 and Eriochrome black T indicator, titrated with 0·1 *M* soln. of complexone III to a steel-blue colour. Magnesium hydroxide is dissolved in excess of buffer soln.; MgO and $MgCO_3$ are dissolved in the minimum quantity of conc. HCl, and the buffer and indicator are then added. Magnesium can be determined in mixtures with Na_2SO_4, NaCl, Na_2CO_3, sucrose or citric acid; the error is ± 0·80 per cent.

E. HAYES

758. **Complexiметric titrations in pharmaceutical analysis. VI. Determination of aluminium.** R. Přibil, J. Čihalik, J. Doležal, V. Simon and J. Zýka (*Českosl. Farmac.*, 1953, **2** [7–8], 223–224; *Referativnýĭ Zh., Khim.*, 1954, Abstr. No. 20,360).—To a sample containing 70 to 130 mg of Al dissolved in 20 ml of water, add 0·1 *M* complexone III soln. (about 2 ml in excess), one drop of methyl red soln. and aq. NH_3 soln. to give a faint yellow colour. Add Eriochrome black T indicator and titrate the excess of complexone III with 0·1 *M* $ZnSO_4$ until the colour changes from greenish-blue to wine-red. In the analysis of powders or tablets, the sample is dissolved by boiling with a little conc. HCl soln.; to the cooled soln. one drop of methyl red is added followed by aq. NH_3 soln. to neutralise; an excess of complexone III is then added and the analysis is completed as above. For pure soln. of Al, the error is −2·7 to + 2·1 per cent., and for pharmaceutical preparations, −3·32 to + 0·4 per cent.

E. HAYES

See also Abstract 726.

Food

759. **Estimation of fermentable sugars.** W. Kempf and E. Lindemann (*Stärke*, 1954, **6**, 217–225).—The method of Pan, Nicholson and Kolachov (*Brit. Abstr. C*, 1953, 2348) for the determination

of glucose, maltose and fermentable oligosaccharides by degradation with bakers' yeast has been examined and is adversely criticised. It was found that the origin and characteristics of the yeast have a variable influence on the reaction and that the time required for complete fermentation of glucose and maltose is greater than that prescribed by the original authors. E. DUX

760. **Determination of sulphur dioxide in molasses.** V. Valter (*Listy Cukrovarn.*, 1953, **69** [1], 9–11; *Referativnýĭ Zh., Khim.*, 1954, Abstr. No. 23,993).—Fit a 750-ml conical flask with a stopper carrying a funnel and an adaptor connected to a condenser and receiver. Add 250 ml of H_2O, 25 ml of 25 per cent. H_3PO_4 and $\simeq$ 0·5 g of pumice, and heat. When steam appears in the condenser, add 4 g of $NaHCO_3$ through the funnel. Dilute a sample of molasses (80 g) with water to 200 ml and remove any ultramarine present by centrifuging. When 20 ml of H_2O have been distilled, fit to the condenser a fresh receiver containing 25 ml of 0·5 *N* iodine soln., 5 ml of 2 *N* H_2SO_4 soln. and 20 ml of H_2O. Add (during 3 min.) 50 ml of molasses soln. to the distillation-flask and distil for 15 min. If there is much frothing, add 10 g of NH_4Cl. Titrate the excess of iodine with $Na_2S_2O_3$. In the presence of NO_3' or when the SO_2 content is $>$ 80 mg per 100 g, the determination is carried out gravimetrically and ultramarine need not be removed. The error is less than 1 mg per 100 g. E. HAYES

761. **Critical evaluation of methods of determining methionine in food proteins.** L. A. Gorozhankina (*Vopr. Pitaniya*, 1953, **12** [4], 71–76; *Referativnýĭ Zh., Khim.*, 1954, Abstr. No. 22,209).—The MacCarthy-Sullivan (colorimetric with Na nitroprusside) and the Levin (iodimetric titration) methods of determining methionine in protein foods are critically examined and modifications of both methods are proposed. The first method is unsuitable for food products that have a high methionine content or contain copper. E. HAYES

762. **Biochemistry of myoglobin. Quantitative determination in beef and pork muscle.** I. D. Ginger, G. D. Wilson and B. S. Schweigert (*J. Agric. Food Chem.*, 1954, **2** [20], 1037–1038).—A spectrophotometric method is described for the determination of myoglobin (**I**) in muscle. *Procedure*—Finely ground muscle (10 g) is mixed with 10 ml of water and kept at 3° to 5° C overnight; the mixture is centrifuged and the supernatant liquid adjusted to pH 7·0. Saturated basic Pb acetate solution (equal to one-quarter of the vol. of the supernatant liquid) is added to precipitate impurities; the mixture is centrifuged, and KH_2PO_4 and K_2HPO_4 are added to the filtered supernatant liquid to bring the phosphate concn. to 3 *M* and the pH to 6·6. The precipitated haemoglobin is removed by filtration; $K_3Fe(CN)_6$ and NaCN are added to an aliquot of the filtrate to give a final concn., after dilution, of 0·6 and 0·8 millimole, respectively. The light absorption of the resulting cyanmetmyoglobin (**II**) is measured at 540 mμ. In calculating the weight of **I** per g of muscle, it is assumed that E for **I** is 11·5 (the same as that for **II**), that the mol. wt. is 16,500 and that **I** is distributed equally between extract and muscle residue. S. C. JOLLY

763. **Polarographic determination of nitrites and nitrates in meat-pickling solutions.** R. Pleticha and E. Křižova (*Průmysl Potravin*, 1953, **4** [9], 383–386; *Referativnýĭ Zh., Khim.*, 1954, Abstr. No. 16,819).—For the determination of nitrate, the method of Kolthoff *et al.* (*J. Amer. Chem. Soc.*, 1944, **66**, 1782) is used. A soln. containing 1 per cent. of the sample together with KCl (0·1 *M*), HCl (0·01 *M*) and uranyl acetate (0·0002 *M*) is polarographed at −1 V. A soln. of KNO_3 (0·001 *M*) is used to prepare a calibration curve. For the determination of nitrite, 9 ml of 10 per cent. acetic acid soln. are placed in the cell and nitrogen is blown through. One ml of a 10 per cent. soln. of the sample, freed from atmospheric oxygen, is then added; the soln. is mixed and then polarographed at −0·6 V, the addition method being used. When nitrate is being determined in spent soln., proteins must first be precipitated with ethanol and then removed by filtration. In the nitrite determination on spent soln., the proteins are pptd. by acetic acid and the use of ethanol is unnecessary. E. HAYES

764. **[Microbiological assay of] essential amino-acids in the milk of Indian buffalo.** N. V. Joshi and H. Raj (*Indian J. Dairy Sci.*, 1954, **7** [3], 139–146).—Assays were carried out on the acid-hydrolysed casein, prepared from the milk and freed from fat and unsaponifiable matter, by the method of Barton-Wright (*Brit. Abstr. C*, 1946, 289). Recoveries of added acids ranged from 93 to 104·5 per cent., and no difference in results was observed when assays were carried out by the above method or by the method as modified by Barton-Wright and Curtis (*Brit. Abstr. C*, 1949, 138). The amino-acid composition of buffalo-milk and cow-milk proteins are very similar. The results conform with previous findings. L. G. L. UNSTEAD-JOSS

765. **Assay of various mould-ripened cheeses for antibiotic activity.** H. H. Wilkowske and W. A. Krienke (*J. Dairy Sci.*, 1954, **37** [10], 1184–1189).—In the method described, an homogenised aqueous dilution (1 + 2) of the sample is adjusted to pH 6·5 to 7·5 with a 0·1 *N* NaOH soln. and then steamed for 5 min. After filtration, the filtrate is diluted to 10 per cent. strength with homogenised milk and then 3 per cent. of an active dairy starter culture is added. Nine-ml quantities mixed with 9 ml of distilled water are at once titrated electrometrically to pH 8·3 with 0·1 *N* NaOH soln., to serve as controls. Nine-ml test portions are incubated for 6 hr. at 30° C, and then similarly diluted and titrated. In the absence of any antibiotic the percentage of lactic acid increases during incubation from about 0·18 to between 0·6 and 0·8, whilst the presence of 0·2 unit or more of penicillin per g of sample suppresses any increase. Fourteen commercial cheeses of four types showed no detectable antibiotic activity. W. H. C. SHAW

766. **Determination of trace elements in table olives and their brines.** A. Vioque and F. A. Pohl (*Grasas y Aceites*, 1954, **5** [3], 111–114).—Traces of heavy metals in table olives and their brines are determined by ashing, removing from salts in higher concn. by $CHCl_3$ extraction with several (successive) organic reagents, concentrating and analysing spectrochemically. *Procedure*—Olive-pulp samples (20 g) are dried to constant weight, ashed at 450° C with the addition of a few drops of conc. HNO_3. Brine (50 ml) is evaporated and the residue is carbonised and incinerated similarly. The ash is dissolved in 10 ml of 6 *N* HCl, diluted to 100 ml, adjusted to pH 3 with 6 *N* HCl or 6 *N* aq. NH_3 soln. and extracted by adding 2 ml of 5 per cent. aq.

Na diethyldithiocarbamate and 15 ml of 0·1 per cent. 8-hydroxyquinoline in $CHCl_3$, shaking and separating the $CHCl_3$ extract, repeating until the $CHCl_3$ is colourless; the solution is adjusted to pH 5, and the extraction is repeated until the extract is again colourless. After adjusting to pH 7, the solution is once more extracted, but 5 ml of 0·01 per cent. diphenylthiocarbazone are added to the other reagents and extraction is continued until the $CHCl_3$ layer remains green. The extracts are combined, evaporated and 10 μl of a 0·1 per cent. Be solution containing 10 per cent. of KNO_3 are added for comparison; the extracts are then calcined at 350° C with a few drops of HNO_3 and the residue is dissolved in 6 *N* HCl and sucked into an automatic levelling microburette with washings of the container to a final vol. of 0·1 ml. Twenty μl of this are analysed spectrographically. The sensitivity varies from 0·02 to 0·5 p.p.m. for different metals and the accuracy is $\pm$ 20 per cent. D. LEIGHTON

767. **Fungicide determination. Quantitative determination of diphenyl in citrus fruits and fruit products by means of chromatostrips.** J. G. Kirchner, J. M. Miller and R. G. Rice (*J. Agric. Food Chem.*, 1954, **2** [20], 1031–1033).—A rapid method for detecting as little as 1 p.p.m. of diphenyl (**I**) in juice, peel, or whole fruit of species of citrus fruit is described. *Procedure*—The sample (1500 ml of juice, or a single fruit ground with 200 ml of water and diluted to 1500 ml, or 100 g of peel ground with 100 ml of water and diluted to 1500 ml) is extracted by boiling gently for 3 hr. in a 3-litre boiling flask fitted to a modified Clevenger distillation apparatus containing 1 ml of *n*-heptane or *iso*octane in the 10-ml receiving burette. The condenser and upper part of the still head are rinsed with 1 ml of solvent; the total vol. of the solvent solution, which contains **I** and citrus oil, is noted. Aliquots are diluted 1 to 10 and 1 to 100 with solvent; 0·01-ml portions of the undiluted and diluted solutions are applied to a series of chromatostrips (Kirchner, Miller and Keller, *Brit. Abstr. C*, 1951, 316), which are developed by ascending capillarity with 1 ml of purified light petroleum (boiling range 30° to 60° C) in stoppered tubes. When the solvent front has moved 5 cm past the origin (approx. 7 min.), the strips are removed, dried in air and examined. In u.v. light, the spot due to **I** is dark against a fluorescent background; the strip in which this spot is just detectable is compared with a similar spot given by known concn. of **I**. Alternatively, strips are developed until the front has moved 10 cm, are then dried in air and a 22-mm long area containing the **I** spot scraped from the full width of the strip into a sintered-glass funnel. The **I** is extracted with ethanol (95 per cent.), the filtrate is diluted to 3 ml, and the u.v. absorption is measured at 248 mμ against a blank of ethanol. Blanks are carried out on the strips and the amount of **I** is derived from a standard curve. The average error is $\pm$ 2·8 per cent. over the range 0·1 to 600 p.p.m. S. C. JOLLY

768. **Determination of sulphur in brewing materials.** P. Weiersmüller (*Schweiz. Brauerei Rdsch.*, 1954, **65** [5], 71–75).—After critical examination of several procedures, the following combination of methods is selected and described in detail. The sample (0·25 to 0·55 g of malt, barley, hops or dried yeast) is oxidised with Na_2O_2 in a micro-bomb by the method of Wurzschmitt (*cf. Brit. Abstr. C*, 1951, 399), the sulphate formed is reduced to sulphide by Roth's method (*Brit. Abstr. C*, 1951, 246) and then determined iodimetrically by Bethge's method (*Anal. Abstr.*, 1954, **1**, 72 and 923). The procedure gives results close to theoretical values for pure S-containing amino-acids, and in the analysis of the above materials it is accurate within $\pm$ 0·020 per cent. P. S. ARUP

769. **A new colorimetric method for determining copper in wine.** M. Ya. Shapiro and V. G. Lapina (*Vinodelie i Vinogradstvo SSSR*, 1953, [6], 6–8; *Referativnyĭ Zh., Khim.*, 1954, Abstr. No. 15,025).—The Cu in a sample is pptd. with $Na_2S_2O_3$ in the presence of H_2SO_4 and the ignited ppt. is dissolved in HNO_3. Traces of Fe are removed by pptn. with aq. NH_3 soln. and the filtrate is evaporated to dryness with HCl and heated to remove ammonium salts; the residue is dissolved in a definite vol. of water. The Cu is then determined colorimetrically; the yellow colour which develops on the addition of 1 per cent. guaiacol and 0·5 per cent. aq. NH_3 soln. is measured. For Cu concn. of 0·1 to 2 mg per litre, the intensity of the colour is proportional to the Cu content; the colour develops fully in 25 min. The reaction can also be used to detect Cu; the sensitivity is 0·04 μg of Cu per ml. Other metallic salts interfere if they are present in amounts 200 to 300 times that of the copper. E. HAYES

770. **The determination of sulphate in wines.** L. Deibner and P. Bénard (*Ind. Agric. Aliment.*, 1954, **71**, 427–436).—The factors which influence the pptn. of the SO_4'' are HCl, excess of precipitant, extent of dilution, various mineral and organic substances, *e.g.*, phosphates, tannin, organic and mineral acids and sugars; these factors are tested on synthetic wines and the results are discussed. An improved method is given for the determination of SO_4'' by means of 2 *N* HCl (5 ml per 100 ml of liquid to be analysed) and 5 ml of a $BaCl_2$ solution containing 100 g of $BaCl_2.2H_2O$ per litre of water. This method may be used for wines containing no free or combined SO_2, and the residue from wines from which sulphite has been removed by distillation with HCl. *Procedure*—Dilute 100 ml of wine to 200 ml with water and add 10 ml of 2 *N* HCl. Boil the liquid and add 10 ml of $BaCl_2$, drop by drop, while stirring the boiling liquid; allow the ppt. to form during 4 hours on an electrically heated plate (Policard) at 60° C, or during two hr. on a bath of boiling water. For small quantities of $BaSO_4$, set the soln. aside overnight. Filtration, washing and heating the ppt. follow the usual routine. S. C. I. ABSTR.

771. **Standards for Indian coffee.** V. Subrahmanyan, D. S. Bhatia, C. P. Natarajan, G. S. Mani, J. R. Iyengar and M. Nagarathnamma (*Curr. Sci.*, 1954, **23** [9], 292–293).—The average chemical composition of forty samples of *Arabica* and twenty of *Robusta* coffee powders is tabulated and the following tentative standard is postulated. Total ash, 3·5 to 4·8 per cent.; water sol. ash, $\not<$ 65 per cent. of total; acid insol. ash, a trace; alkalinity of soluble ash, 30 to 47 ml of *N* HCl per 100 g; water extract (Jones method), 26 to 33 per cent.; caffeine, $\not<$ 1·0 per cent.; total *N*, 2·2 to 3·1 per cent. E. G. BRICKELL

772. **Identification of some of the natural constituents of vanilla. Chromatographic separation of "ethylvanillin".** S. Stoll and Y. Bouteville (*Ann. Falsif.*, 1954, **47**, 183–187).—A chromatographic method for the examination of commercial vanilla is given, and the detection of "ethylvanillin"

is discussed. The vanilla is extracted with ethanol and the extract is evaporated, the ethanol being continuously replaced with water. The aqueous soln. is cleared with basic lead acetate. The clear liquid is extracted with ether. The ethereal solution, in turn, is extracted with aq. NH_3 soln., which is subsequently acidified and re-extracted with ether. The two ether extracts are then each examined by ascending chromatography, using 10 to 20 μg of the solid residue, and with butanol as developer. The various constituents of the vanilla are identified by treating the chromatogram with the reagents given below. Vanillic acid (R_F 0·12); *reagent*—saturated aq. *p*-nitroaniline (1 vol.) mixed with 1 per cent. aq. $NaNO_3$ soln. (1 vol.). The chromatogram is subsequently treated with sat. aq. Na_2CO_3. Coumarin (R_F 0·94); *reagents*—(1) 1 per cent. ethanolic KOH; (2) as under vanillic acid, after a pre-treatment with sat. aq. Na_2CO_3. Vanillin (R_F 0·48); *reagents*—(*i*) NH_3 (gaseous); (*ii*) saturated 2:4-dinitrophenylhydrazine in 2 *N* HCl. Piperonal (R_F 0·96); *reagent*—as used for vanillin. "Ethylvanillin" (R_F 0·63); *reagent*—NH_3 (gaseous) followed by hydrazine sulphate (Dingemans *Chem. Weekbl.*, 1930, **27**, 694). All chromatograms should be examined by ordinary and by u.v. light. The last reagent distinguishes between "ethylvanillin" and a naturally occurring substance, not previously recognised, which has the same R_F value as "ethylvanillin" and has been confused with the latter.

G. B. THACKRAY

773. **Detection of vegetable fats and oils by means of the phytosteryl acetate test.** H. Hadorn and R. Jungkunz (*Mitt. Lebensm. Hyg., Bern*, 1954, **45** [5], 389–396).—An improved procedure is described. The unsaponifiable matter from 20 g of the oil or fat, dissolved in 10 ml of acetone, is treated with $\geq$ 10 ml of ethanolic 1 per cent. digitonin. The digitonides, obtained after evaporation, are digested for a short time in hot water, filtered off, washed (separately) with acetone, ether, $CHCl_3$ and ether again and then acetylated by boiling with 3 ml of acetic anhydride. The resulting soln., after filtration (hot) and cooling, deposits crystals of the sterol acetates which, after washing with 90 per cent. ethanol at 0° C, are sufficiently pure for the satisfactory determination of the m.p. without recrystallisation.

P. S. ARUP

774. **Estimation of dihydroxystearic acid in castor oil.** N. R. Kamath, J. G. Kane and B. Sreenivasan (*Current Sci.*, 1954, **23** [8], 262).—Preliminary details are given of a method which involves periodic acid for the determination of dihydroxystearic acid in castor oil. Interfering unsaturated acids are destroyed by hydrogenating the castor oil in ethanolic solution to a low I value (6 to 8) by means of Raney nickel (two 3 per cent. portions added at intervals of 5 hr.) at 30° to 33° C under a pressure of 40 to 42 lb per sq. in. of H. To avoid interference from possible mono- and diglycerides, the methyl esters of the hardened oil are formed; 1 to 2 g of the esters are dissolved in 10 ml of glacial acetic acid and treated with 5 ml of 0·025 *N* periodic acid in glacial acetic acid at 15° to 20° C. After 10 min., 25 ml of 10 per cent. aq. KI solution and 10 ml of $CHCl_3$ are added and the liberated iodine is titrated with 0·1 *N* $Na_2S_2O_3$; a blank determination is carried out. Results for castor oil (1·8 to 2·7 per cent.) are higher than those previously reported (0·6 to 1·3 per cent.), but are probably more representative.

S. C. JOLLY

775. **Determination of fat peroxides in the presence of phospholipids.** L. Hartman (*J. Sci. Food Agric.*, 1954, **5** [10], 476–481).—The interference caused by phospholipids in the ferric thiocyanate method of peroxide estimation is avoided in this new colorimetric method. The fat is extracted as in the Hills - Thiel procedure for milk powder (*J. Dairy Res.*, 1946, **14**, 340), by refluxing the sample with 15 to 20 ml of benzene - methanol mixture (7:3) at 60° C for 5 min. The extract is filtered into a narrow test tube calibrated at 8 ml. One ml of $FeCl_2$ reagent [1 ml of a stock solution of $FeCl_2$ (0·8 g of $BaCl_2.2H_2O$ and 1 g of $FeSO_4.7H_2O$ dissolved in 100 ml of water acidified with 2 ml of 10 *N* HCl and then filtered) added to 20 ml of benzene - methanol mixture (1:1) just before use] is added, the mixture is shaken, and, after 10 min. at room temperature, 1 ml of dichlorophenolindophenol solution (**I**) (0·04 g of commercial dye dissolved in 10 ml of methanol and diluted to 100 ml with benzene) is added, followed by 1 drop of pyrophosphoric acid solution (**II**) (10 g dissolved in 100 ml of methanol). The light absorption of the solution is measured immediately against a blank, prepared by using 8 ml of benzene - methanol mixture instead of fat solution. **I** is standardised as follows: 15 ml of $FeSO_4$ solution (1 g of $FeSO_4.7H_2O$, reagent grade, dissolved in water containing 2 ml of conc. HCl, and diluted to 100 ml) are diluted with methanol to give a concn. of 56 μg of Fe^{++} in 1 ml of solution. To 1 ml of this solution, 1 ml of **I** is added, followed by 1·9 ml of methanol and 1 drop of **II**, and the mixture is diluted to 10 ml with benzene. A blank is prepared from 2 ml of the diluted $FeSO_4$ solution, 1 ml of **I**, 0·9 ml of methanol and 1 drop of **II**, and the mixture is diluted to 10 ml with benzene; if the colour is not pink, the concn. of **I** must be adjusted. From the colorimeter reading, E corresponding to 55·84 μg of Fe^{+++} ($\equiv$ to 1 micro-equiv. of peroxide O) can be calculated; in the work reported, E at 530 mμ was 0·500 (1-cm cell).

S. C. JOLLY

776. **Antioxidants in fats and oils. II. Testing of fats and oils for antioxidants by means of the Swift stability test.** F. Wenger (*Mitt. Lebensm. Hyg., Bern*, 1954, **45** [5], 364–383).—A modified Swift stability testing assembly is described in which the reaction temp. and the speed of the current of air or O can be varied. When working at 115° C with a current of 2·33 ml of O per sec., the presence of antioxidants in lard or arachis oil can be presumed, when the peroxide value reached after 2 or 6 hr., respectively, is < 50.

P. S. ARUP

777. **Antioxidants in fats and oils. III. Simple tests for antioxidants.** F. Wenger (*Mitt. Lebensm. Hyg., Bern*, 1954, **45** [5], 383–388).—For each of the following three tests, 1 g of the oil or fat is dissolved by heating it in 2 ml of absolute ethanol, the ethanolic layer formed on cooling is used for the tests. The presence of $\simeq$ 0·01 per cent. of gallates, nordihydroguaiaretic acid (**I**) or guaiacum is indicated by the decoloration, during 2 to 3 min., of a reagent made by diluting a (1 + 1) mixture of aq. 0·2 per cent. $FeCl_3$ and 0·1 *N* NH_4CNS with an equal volume of water. The test can be made roughly quant. Butylated hydroxyanisole ($\leq$ 0·001 per cent.), which is not detected by the previous reaction, can be detected by the blue coloration formed with 2:6-dichloroquinonechlorimine and borax (*cf.* Mahon and Chapman, *Brit. Abstr. C*, 1952, 388), but the reaction is rendered less distinct by the simultaneous presence of **I** or gallates. Gallates are detected by the characteristic red coloration given with conc.

aq. NH_3 soln. A specific test for **I** is in course of publication. P. S. ARUP

778. **The determination of L-ascorbic acid in food products.** J. Blattná, J. Fránger, V. Šanda, P. Zuman and D. Žuffova (*Průmysl Potravin*, 1953, **4** [9], 402–408; *Referativnýĭ Zh., Khim.*, 1954, Abstr. No. 15,069).—A volumetric and a polarographic method are described. In the volumetric method, 10 to 20 ml of a solution or extract are made up to 50 ml with a mixture of acetic acid and HPO_3 (15 g of solid HPO_3 dissolved in 40 ml of glacial acetic acid and 200 ml of water; the soln. is diluted to 500 ml, filtered and set aside in the cold), and titrated with 0·001 *N* aq. dichlorophenolindophenol until a rose colour persists for 15 min. The minimum amount of ascorbic acid that can be determined is 25 to 50 μg and the limits of error are ± 10 per cent. In the analysis of coloured substances, the titration is carried out in the presence of xylene. and the colour change is noted in the xylene layer. When the material contains SO_2, acetone is added; reducing substances are allowed for by carrying out a blank estimation after the ascorbic acid has been condensed with formaldehyde. In the polarographic method, the ascorbic acid is polarographed in an acetate buffer at pH 4·7 (Kodichek and Wenig, *Nature*, 1938, **142**, 35), and the increment method is used. E. HAYES

779. **Cleaning culture tubes for the U.S.P. vitamin B_{12} microbiological assay.** E. M. Stapert and L. Stubberfield (*J. Amer. Pharm. Ass., Sci. Ed.*, 1954, **43**, 382).—The use of tubes cleaned with 65 per cent. v/v H_2SO_4 eliminates a serious inconsistency in the assay of vitamin B_{12}. J. CALEY

Sanitation

780. **Direct field determination of lead in air.** M. O. Amdur and L. Silverman (*Arch. Ind. Hyg.*, 1954, **10** [2], 152–157).—At pH 2·79, most lead compounds give a coloured complex with tetrahydroxyquinone; tetraethyl-lead fails to react. The reagent in alcoholic solution is stable for some months at room temp. Air containing Pb is drawn through a confined spot test paper (100 sq. mm), the reagent and buffer are applied, and the colour produced is compared with known standards. Attempts to make the test more accurate by using a densitometer were unsuccessful. Cd interferes with the test if present in larger quantities than the Pb. C. E. SEARLE

781. **Determination of the total hardness of drinking water by the Versenate method.** M. W. E. Evers and J. S. N. Cramer (*Chem. Weekbl.*, 1954, **50** [12], 218–219).—Errors in the method of Sijderius (*Brit. Abstr. C*, 1952, 369) are avoided by the following procedure. Bicarbonate is destroyed by titration, and the indicator is removed by adding bromine. After heating on the water-bath for 30 min. and then cooling to 40° C, 4 ml of buffer solution (0·891 per cent. of NH_4Cl, 9·46 per cent. of aq. NH_3 soln. (sp. gr. 0·88), and 0·6 per cent. of magnesium ethylenediaminetetra-acetate) and 6 drops of Eriochrome black T soln. are added. The mixture is titrated with 1 per cent. sodium ethylenediaminetetra-acetate solution that has been standardised against calcium carbonate. G. MIDDLETON

782. **Determination of oxygen dissolved in water by ascorbic acid.** L. Erdey and F. Szabadváry (*Acta Chim. Hung.*, 1954, **4** [2–4], 325–332).—A flask of known vol. is filled with water. With a pipette, 2 ml of aq. NaOH (1 pt. NaOH and 2 pt. H_2O) are brought into the bottom of the flask, followed by 2 ml of 20 per cent. $(NH_4)_2Fe(SO_4)_2$, which has been reduced by Cd or Fe powder. (If the solution has not been reduced, a blank determination of $Fe^{\cdots}$ with ascorbic acid is necessary; this titre must be deducted from the vol. of the acid used in the final titration.) The flask is closed with a moistened glass stopper, shaken and shielded from the air until the pptd. iron hydroxide has subsided. The ppt. is then dissolved in 5 ml of dil. HCl (2 + 1) or 20 ml of 25 per cent. $NaHSO_4$ introduced by a pipette. The solid indicator mixture (2 to 3 mg of a mixture of 1 part of Variamine blue and 9 parts of NaCl) is added; the flask contents are warmed to 40° to 50° C and titrated with 0·01 *N* ascorbic acid until the violet colour disappears. Winkler's determination is modified by adding $FeSO_4$ containing Mn to the oxidised solutions and titrating the resulting $Fe^{\cdots}$ with ascorbic acid, or by adding a known amount of ascorbic acid and determining the excess with $FeCl_3$. The first procedure is accurate but not the second. H. WREN

783. **A turbidimetric sulphate determination.** J. F. Thomas and J. E. Cotton (*Water & Sewage Wks.*, 1954, **101** [10], 462–465).—A quick method of sulphate determination, specifically intended for waters containing low sulphate concentrations, is described. After the usual barium sulphate precipitation, the turbidity is determined at 380 mμ and compared with a standard. Beer's law is followed between 20 and 80 p.p.m. of SO_4''. Standards are given. A. WEBSTER

784. **The effect of sunlight on iodimetric titrations [on sewage].** T. Stones (*J. Inst. Sewage Purif.*, 1952, [4], 415–416).—In the determination of oxygen absorption from acid $KMnO_4$ by samples of sewage, more I is liberated from the KI in sunlight. Sunlight probably promotes liberation of I from KI in the presence of acid and dissolved oxygen. A. WEBSTER

785. **The analysis of *pp'*-dichlorodiphenyltrichloroethane (DDT).** S. Wolf (*Z. anal. Chem.*, 1954, **143** [6], 415–422).—*pp'*-Dichlorodiphenyltrichloroethane (**I**) can be assayed in the presence of the *op'*-isomer by determination, at 30° C, of the dielectric constant of a solution in dichlorobenzene; the error is ≃ 2 per cent. A more accurate (≃ 1 per cent.), but less rapid, gravimetric procedure is also described; the sample is extracted at 50° C with a saturated solution of **I** in 75 per cent. ethanol, and the residue is dried at 80° C and weighed. A. R. ROGERS

786. **The assay of "pyrethrin" and allethrin concentrates with 2:4-dinitrophenylhydrazine.** B. P. Moore (*J. Sci. Food Agric.*, 1954, **5** [10], 500–503).—A consistent and accurate colorimetric method is described for the assay of "pyrethrin" and allethrin concentrates. *Reference curve*—To 20 to 60-mg portions of cryst. α-(±)-*trans*-allethrin (obtained from commercial allethrin by cooling and recrystallising from light petroleum to constant m.p. of 50·5° to 51° C) dissolved in 15 ml of 10 per cent. v/v H_2SO_4 in carbonyl-free methanol, 80 mg of 2:4-dinitrophenylhydrazine (**I**) are added. The mixture is heated at 80° C for 20 min. in a closed flask, cooled, transferred with 20 ml of ether to a separator and extracted with 15, 15 and 10-ml portions of water. The combined aq. extracts are extracted with two

20-ml portions of ether, each extract being washed with two 10-ml portions of water. The combined ethereal solutions are evaporated to low bulk, diluted with 25 ml of dry benzene, evaporated further until the benzene begins to distil, cooled, and diluted with an equal vol. of dry light petroleum (boiling range 40° to 60° C), and the mixture is chromatographed on a column (diam. 2·5 cm) of alumina, grade III (50 g), prepared by adding it as a slurry in a benzene - light petroleum mixture (1 + 1). The chromatogram is developed with the solvent mixture until the orange allethrin derivative nears the bottom of the column, when it is eluted with a benzene - light petroleum mixture (2 + 1). The bulk of the light petroleum is evaporated off *in vacuo* from the eluate, which is then diluted to 100 ml with dry benzene and a 10-ml aliquot is diluted to 100 ml with dry benzene. The optical density is measured against benzene in a suitable photometer, using a 1-cm cell and a blue-green filter, and the results are plotted graphically. *Assay of concentrates*—Quantities of concentrates containing approx. 40 mg of active material are treated as described above, but with 120 to 150 mg of **I**, a large excess being particularly necessary with crude "pyrethrin" concentrates. Polymerised material remains at the top of the column above the **I** band and other impurities pass through rapidly as a pale-yellow fore-run. Allethrin equiv. are obtained from the reference curve and are converted into pyrethrin equiv. by multiplying by 1·07. Results obtained by this method are lower than those obtained by the A.O.A.C. method.

S. C. Jolly

See also Abstracts 567, 812.

Agriculture and Plant Biochemistry

787. **Separations of polysaccharides related to starch by electrokinetic ultrafiltration in collodion membranes.** D. L. Mould and R. L. M. Synge (*Biochem. J.*, 1954, **58** [4], 571–585).—A method is described for chromatography inside a strip of collodion ultrafiltration membrane. Flow of liquid in the membrane is produced electrokinetically. Compact zones with definite R_F values are obtained with enzymically synthesised polysaccharides. The method can be used to characterise linear dextrins according to mol. wt., and to estimate the degree of polymerisation range for linear polymers.

J. N. Ashley

788. **The electrophoretic mobility and fractionation of complexes of hydrolysis products of amylose with iodine and potassium iodide.** D. L. Mould and R. L. M. Synge (*Biochem. J.*, 1954, **58** [4], 585–593).—An apparatus is described for continuous electrophoretic fractionation of α-amylase-hydrolysed amylose in agar jelly in the presence of iodine in KI. Separation is effected into zones of various colours depending on the degree of polymerisation. The method may be useful in the fractionation of polydisperse substances, such as amylopectin or glycogen. J. N. Ashley

789. **Comparative study of methods for determination of pentosans.** E. L. Raekelboom and J. R. Istas (*Ind. Chim. Belge*, 1954, **19** [9], 915–922).—A review is presented (32 references). Neither the classical method of distillation with 12 per cent. HCl, nor the method of Jayme and Sarten (*Biochem. Z.*, 1941, **308**, 109) involving distillation with 40 per cent. HBr, gives satisfactory results with mixtures of sugars or with wood. The various reagents used for the determination of the furfural produced give inconsistent results. Until the mechanism of the conversion of pentosans to pentoses and furfural is more clearly known, a search for new methods of hydrolysis and for determination of furfural is worthless. S.C.I. Abstr.

790. **Microchemical determination of uronic acids in the presence of sugars.** L. Federico and M. Ciucani (*Chim. e Ind.*, 1954, **36** [8], 598–603).—Several methods have been examined; the carbazole method gives good results when the ratio of uronic acid to sugar is > 1 to 5; galactose interferes more than glucose or pentoses. In the orcinol method for determining uronic acid and pentoses, glucose and galactose do not interfere except at high concn., but fructose, if present in an appreciably greater amount than the pentose or uronic acid, invalidates the method. The Tracey method for determining pentoses, based on the colour reaction with aniline and acetic and oxalic acids, is satisfactory, if the ratio of pentoses to glucose or fructose is > 1 to 5 and of pentoses to galactose or uronic acid is > 2 to 5. The behaviour of the various compounds in the anthrone colour reaction has been examined; glucose and fructose give strong colorations, galactose, galacturonic acid, xylose and arabinose rather weaker ones and glucuronic acid practically none. A method for the quant. separation of uronic acid from sugars by absorption on Amberlite IRA-400, with a modification of the technique of Khym and Doherty (*J. Amer. Chem. Soc.*, 1952, **74**, 3199), is described. L. A. O'Neill

791. **Determination of uronic acid in alduronic acids, polysaccharides and oxycelluloses.** A. Meller (*Svensk Papperstidning*, 1954, **57**, 741–744).—The quantitative determination is based on an evaluation of kinetic data obtained by measuring the rate of CO_2 evolution from a homogeneous H_2SO_4 soln. of the materials. The carboxyl group content of uronic acids may be determined with an accuracy of ± 4 per cent. S. V. Sergeant

792. **Studies in triterpenoids. II. Colouring reagent for triterpenoids in paper chromatography.** T. Koyama and K. Kubota (*Kumamoto Pharm. Bull.*, 1954, [1], 44–46).—The Liebermann reaction for triterpenoids can be used on paper chromatograms if the proportion of conc. H_2SO_4 and acetic anhydride is 0·6 to 1 by vol. This reagent will detect 3 μg of ursolic acid on a paper chromatogram. Separation of ursolic acid from oleanolic acid on paper was found to be impracticable with paper impregnated with silicic acid, owing to the very similar R_F values of the two acids.

S.C.I. Abstr.

793. **A colorimetric procedure for the determination of tryptophan and tyrosine in the juice expressed from potatoes.** Th. Breyhan (*Z. anal. Chem.*, 1954, **143** [4], 241–244).—To the potato juice (1 vol.), add 96 per cent. ethanol (4 vol.), shake for 10 min. and filter. To an aliquot (1 ml) of the filtrate, add 10 per cent. acid $HgSO_4$ soln. (2 ml), set aside for 60 min. and filter; the filtrate contains the tyrosine. Wash the precipitate, which contains the tryptophan, and dissolve it in 0·8 per cent. KCN soln. (2 ml), add 10 per cent. trichloroacetic acid (1 ml), dilute to 10 ml, shake, set aside for 10 min. and filter. Take 5 ml of the filtrate, cool to 0° C, add 1 per cent. $NaNO_2$ soln. (0·1 ml) and measure the extinction at 436 mμ from 7 to 10 min. later. Dilute the filtrate and washings containing the tyrosine to 10 ml, shake and filter. To 5 ml of the filtrate, add

1 per cent. $NaNO_2$ soln. (0·1 ml), shake and, after 30 min., measure the extinction at 578 mμ. Compare the readings obtained with a calibration graph constructed with known amounts of tyrosine and tryptophan in 0·5 per cent. glucose soln. An accuracy of 7 to 10 per cent. is claimed.

P. S. STROSS

794. **Photometric method for the micro-determination of copper in soil.** L. N. Lapin and V. P. Makarova (*Pochvovedenie*, 1953, [8], 82–89; *Referativnyĭ Zh., Khim.*, 1954, Abstr. No. 22,146).—The method is based on the reaction of Cu with diphenylcarbazone (**I**). *Procedure*—Organic matter in a sample of finely ground soil (500 mg) is destroyed by heating first with a mixture of conc. H_2SO_4 (5 ml) and K_2SO_4 (2 g) and then with 1 ml of 30 per cent. aq. H_2O_2. Aqueous NH_3 (sp. gr. 0·92) is added to the residue until the soln. is neutral to phenolphthalein; after the addition of a 2-ml excess, the soln. is made up to 50 ml with water. To 10 ml of the clear supernatant liquid, 0·5 ml of an ammoniacal soln. of phenolphthalein (10 mg of phenolphthalein, 1 ml of conc. aq. NH_3 and 9 ml of water) are added and then dil. H_2SO_4 (1 + 4), dropwise, to give a pale-rose colour. Crystalline KH_2PO_4 (0·5 g) is added to make the pH 4 to 5 and the vol. is made up to 15 ml. Five ml of a soln. of **I** (10 mg of **I** dissolved by heating on the water-bath in 30 to 40 ml of benzene is cooled and made up to 100 ml; the addition of 0·4 ml of freshly distilled pyridine ensures more rapid and complete development of the complex) are added. The colour of the benzene soln. is measured in a step-photometer. The complex has max. at 528 mμ and the extinction obeys Beer's law. The complex contains ≃ 12 per cent. of Cu, is insoluble in H_2O, slightly soluble in pentanol, ether and CCl_4, and soluble in benzene and its homologues. The error does not exceed 0·7 per cent.

E. HAYES

795. **Report of the Seed and Meal Analysis Committee, 1953–1954.** (*J. Amer. Oil Chem. Soc.*, 1954, **31** [9], 370–373).—The reports and recommendations of two sub-committees are presented. Methods for sampling and for the determination of moisture plus volatile matter and oil in linseed, and of moisture, plus volatile matter, oil, protein, ash and crude fibre in linseed meal are investigated and some modifications recommended. Methods for automatic sampling of bulk oilseed meals are compared with probe sampling, and it is recommended that a procedure be included in A.O.C.S. official method Ba 1–38 for the automatic sampling of oilseed meals at the time of loading for bulk shipments. A brief outline of such a method is given.

D. BAILEY

796. **Fungicide analysis. Direct determination of methyl mercuric dicyandiamide.** D. Polley and V. L. Miller (*J. Agric. Food Chem.*, 1954, **2** [20], 1030–1031).—A colorimetric method is described for determining 1 to 100 μg of methylmercuridicyandiamide (**I**) or chloride in aq. soln. with an accuracy of ± 3 per cent. or ± 2 μg, whichever is greater. *Procedure*—A 2-ml aliquot of an aq. soln. containing 15 to 70 μg of **I** is added to 15·0 ml of $CHCl_3$ and 20 ml of hydroxylamine reagent (1 ml of a 20 per cent. hydroxylamine hydrochloride solution added to 400 ml of a solution that is *N* with respect to NaCl and 0·05 *N* to HCl; the substances are dithizone extracted). The mixture is shaken vigorously for 1 min. and the $CHCl_3$ layer is added to 20 ml of dithizone-extracted buffer solution (77 g of trihydrated ammonium acetate in 1 litre of water adjusted to pH 6·0 with acetic acid); the extraction is repeated with 5·0 ml of $CHCl_3$ and the extract is added to the buffer solution. To the buffer - $CHCl_3$ mixture, 1 ml of dithizone solution [1 mg per ml of $CHCl_3$, diluted (4 + 50) with $CHCl_3$ just before use] is added and the mixture is shaken for 30 sec.; the light absorption of the $CHCl_3$ layer is determined in a photo-electric absorptiometer with a suitable filter. The content of **I** is derived by reference to a standard curve prepared from a solution containing 100 μg of **I** per ml. Amounts < 1 mg of Cu, Co, Cd, Fe, Pb, Ni, Ag, Zn, Bi and Hg^{++} do not interfere.

S. C. JOLLY

5.—GENERAL TECHNIQUE AND LABORATORY APPARATUS

General

797. **Continuous gas-analysis apparatus.** H. Engelhardt and H. Bönnhoff (*Chem. Rundschau*, 1954, **7** [12], 222–223).—An apparatus is illustrated and described.

H. WREN

798. **Simplified construction of a straight-through metal O-ring-sealed vacuum stopcock.** G. W. Green (*J. Sci. Instrum.*, 1954, **31** [12], 473).—The stopcock consists of a brass body and duralumin plug, gas-flow being diametrical; opening and closing is accomplished by rotation of the plug. Gas-tight seals are formed by O-ring gaskets fitting between the plug and barrel, and also round the inlet and exit ports.

G. SKIRROW

799. **A simple valve to prevent back suction in a vacuum system.** M. C. Chaco (*J. Chem. Educ.*, 1954, **31** [10], 547).—Details and dimensions are given for the construction of a simple valve to prevent back suction in a water-pump vacuum system.

A. LEDWITH

800. **Characteristics of some modern analytical filter-"papers."** A. Grüne (*Chem.-Ing.-Tech.*, 1954, **26** [4], 221–224).—A review is presented of cellulose-based and plastic-based filters produced by Schleicher and Schüll. Figures are quoted characterising the filtration rates and retentivities and resistance to solvents and acid and alkali for the various grades. Glass-fibre filter-"papers" are also dealt with.

R. C. MURRAY

801. **Column-electrophoresis apparatus.** Anon. (*Sci. Tools*, 1954, **1**, 19).—An apparatus for column electrophoresis or electrochromatography is described. The capillary column is packed by sedimentation with starch, cellulose fibres or similar material for pure electrophoresis, or, when subsequent chromatography is to be carried out, with any sorbent.

A. M. SPRATT

802. **A simple device for the rapid evaporation of chromatographic samples.** J. Kahan (*J. Lab. Clin. Med.*, 1954, **44** [4], 661–662).—The apparatus described consists of a rack for ten test tubes, over which is mounted a removable system of pipes for delivering compressed air on to the surface of the solvent in the tubes. With the tubes partly immersed in a water-bath, up to 50-ml amounts of any solvent boiling between 40° and 160° C can be evaporated rapidly.

W. H. C. SHAW

803. **A method for varying the rate of solvent flow in downward paper chromatography.** S. R. Finn and J. W. James (*Chem. & Ind.*, 1954 [41], 1252–1253).—The rate at which a solvent is applied to a chromatographic sheet is restricted by its passage through a reservoir containing a pad of filter-paper strips, being supplied from a trough by paper wicks. This method allows the chromatographic sheet to be supplied at a variable rate, since the number, size and grade of paper wicks are variable. The reservoir provides a uniform solvent front, and eliminates "scolloping." G. R. WHALLEY

804. **Paper chromatography of cations with azo derivatives of 8-hydroxyquinoline.** Q. Fernando and M. de Silva (*Analyst*, 1954, **79**, 711–713).—Some of the azo derivatives of 8-hydroxyquinoline give intense colours with metallic ions and are, in this respect, better suited than 8-hydroxyquinoline for paper chromatography; they are also more selective than some previously investigated derivatives. R_F values for several cations were determined on paper impregnated with six azo derivatives of 8-hydroxyquinoline, by the use of ethanol and methanol as developing solvents. 5-*p*-Tolylazo- and 5-*p*-acetylaminophenylazo-8-hydroxyquinoline did not permit migration of the cations studied; 7-phenylazo-8-hydroxyquinoline-5-sulphonic acid permitted migration of the cations to almost the same extent. 5-*o*-Carboxyphenylazo- and 5-*p*-sulphamylphenylazo-8-hydroxyquinolines seem to be suitable for cationic separations.

A. O. JONES

805. **A laboratory mechanical shaker.** P. D'Ath and E. Smart (*Chem. & Ind.*, 1954, [41], 1253–1254).—An electrically driven variable shaker capable of simultaneously shaking 20 to 30 250-ml flasks is described. G. R. WHALLEY

806. **Distillation flask for continuous processes.** J. Pinkava (*Chem. Listy*, 1954, **48** [3], 455–457).—The 20-ml still for continuous distillation described is especially suitable for the atmospheric or vacuum distillation of foaming liquids. The liquid is boiled in an electrically heated side-arm, from where it flows tangentially into the flask.

G. GLASER

807. **Ground-glass joints leakproof to corrosive chemicals.** L. D. Quin and R. B. Greenlee (*J. Chem. Educ.*, 1954, **31** [10], 518).—A joint sealed with molten Saran (a vinylidene chloride polymer) is resistant to leaking for periods up to 240 hr., when used for refluxing with corrosive chemicals such as chlorosulphonic acid, sulphuryl chloride, thionyl chloride, fuming nitric acid, phosphorus oxychloride, phosphorus trichloride, phosphorus tribromide and various organo-phosphorus halides. The seal is effected by applying the polymer, in the molten state, to the male joint and rapidly inserting it into the female joint, turning several times to obtain a uniform coat. The sealed joint is reopened by warming with a flame and separated when the plastic softens. The seal can be applied to nearly all ground-glass equipment and can be used at both atmospheric and sub-atmospheric pressures. The seal is also effective above the softening point of the plastic. A. LEDWITH

808. **A capillary viscometer (Zeitfuchs Cross-arm type) for series investigations of small quantities of liquids.** H. de Miranda (*Chem. Weekbl.* 1954, **50** [43], 741–742).—The Zeitfuchs viscometer is modified by bending the outlet tube round, making it easier to suck dry, and surrounding it with a tubular water jacket. Only 0·5 ml of liquid is required for a determination (*cf.* Johnson *et al. Brit. Abstr. C*, 1953, 102). G. MIDDLETON

809. **Precision laboratory standards of mass and laboratory weights.** T. W. Lashof and L. B. Macurdy (U.S. Dep. Comm. Nat. Bur. Stand. Circ. 547, Sec. 1, 24 pp.).—This publication, which forms part of a series that is to replace the circular issued in 1918 on the design and testing of standards of mass, comprises the specifications of the National Bureau of Standards for precision laboratory standards and other laboratory weights, together with regulations governing the submission of these weights to the Bureau for test. There is a new class (J) of metric weights designed as standards for weighing equipment used in the precise determination of small masses in ultramicro-analysis, and this is followed, in order of decreasing precision, by class M (high precision scientific standards for semi-micro and microchemical work), class S (for high-precision analytical work) and class S–1 (routine analytical work). The denominations, composition, construction, marking, as well as the tolerances, are described for the weights of each class. J. M. JACOBS

810. **Portable liquid-barrier equipment for sampling airborne dust over prolonged periods.** C. B. Barker, D. T. O'Connor and G. E. Winder (Minist. Fuel Pwr. Safety Min., 1954, Res. Rep. 93, 18 pp.).—The simple portable dust-sampling apparatus described can be used to collect a sample over a whole shift (so that a gravimetric estimate of dust concn. can be obtained) or over several shifts, for compositional analysis. An aspirator unit (driven by compressed air) draws a sample of dust-laden air, at a controlled rate, through a liquid dust-trap. The apparatus is similar in principle to the U.S. Bureau of Mines midget impinger, but is more robustly designed for use underground. Pentyl, hexyl and benzyl alcohols have been used in the collecting flask and proved satisfactory. The collecting efficiency of the apparatus is discussed.

J. M. JACOBS

811. **The preparation of pressed discs of purified potassium chloride containing solid samples for infrared spectrometry.** J. L. Hales and W. Kynaston (*Analyst*, 1954, **79**, 702–706).—To avoid certain difficulties in making pressed discs of KBr for infrared spectrometry, a method of preparing finely divided pure salt by pptn. was developed; KCl, with its lower chemical reactivity and higher stability to heat, was used in preference to KBr. Distilled water is saturated at room temp. with analytical-grade KCl and the soln. is filtered. Three-quarters of the soln. is stirred into an equal vol. of ice-cold pure conc. HCl. The pptd. salt is collected on, and compressed into, a sintered-glass funnel fitted to a Buchner flask; it is allowed to drain under suction. The remainder of the original soln. is used in two portions to wash the salt. The product is pressed in filter-paper and then heated at 120° C in a porcelain or silica dish for several hours. The resulting cake, broken into small pieces, is heated in an electric muffle furnace at 400° to 450° C to remove HCl and water. Complete removal of HCl is indicated by absence of absorption at 3 to 5 μ and 8 to 10·5 μ. The final product yields glass-clear discs at a pressure of 35 tons per sq. in. without any

previous grinding. The construction and performance of a simple electrical micro-balance for weighing the small amounts of solids to be added to the disc is described. A. O. JONES

812. **A simple laboratory apparatus for the continuous treatment of waste liquors with activated sludge.** P. G. Fohr and E. Kagei (*Chem. Weekbl.*, 1954, **50** [42], 726–727).—The apparatus for experimental biological purification of waste liquor consists of an aspirator bottle with a stirrer which causes the liquid to rotate, leaving a hollow cone in the centre of the surface. A sedimentation tube is attached to the tubulure and a syphon tube leads back from the bottom of this tube to the hollow centre of the liquid surface in the aspirator. There is also a constant level overflow from the side tube. This arrangement gives good aeration and no frothing. G. MIDDLETON

813. **Determination of the fine-dust content of air.** D. Hasenclever (*Chem.-Ing.-Tech.*, 1954, **26** [4], 180–187).—A review is given of the principal instruments used in Germany for determining the fine-dust content of air. Special reference is made to the range of application of thermal precipitator, HS Konimeter, Midget impinger, Tyndallometer and Gothe and Martius filter devices.

R. C. MURRAY

See also Abstracts 657, 689, 731, 788.

Optical

814. **Simple microscope attachment for observing high-temperature phenomena.** J. H. Welch (*J. Sci. Instrum.*, 1954, **31** [12], 458–462). The material to be examined is held in a loop at the tip of a small thermocouple, which is heated by a 50-c.p.s. supply. A vibrating switch permits the heating current to flow only during alternate half-cycles, the measuring circuit being used during the intermediate half-cycles. At the max. temp., 1750° C, the power dissipation is about 6 W. Optical observation is made with a 25-mm objective, although a 16-mm objective can be used without special precautions.

G. SKIRROW

815. **Direct-reading apparatus for emission spectral analysis.** E. Lüscher (*Chem. Rundschau*, 1954, **7** [18], 340–342).—The theory of spectral analysis is developed. Accounts are given of Orsag's spectrolecteur, Baird's direct-reading spectrometer and various types of quantometer. The applicability of spectral analysis in industry is illustrated with reference to a steel works.

H. WREN

816. **Micro-photometer registering absolute values of photographic emulsion blackening.** František Khol (*Slaboproudý Obzor*, 1953, **14** [6], 280–283; *Referativnyĭ Zh., Khim.*, 1954, Abstr. No. 24,023).—A micro-photometer (micro-densitometer) that automatically registers absolute values for the blackening of a photographic emulsion is described. It permits the rapid evaluation of spectral-line intensity and can be used in daylight.

E. HAYES

See also Abstract 553.

Electrical

817. **Observations on an automatic titrimeter.** J. Haslam and D. C. M. Squirrell (*Analyst*, 1954, **79**, 689–696).—An account is given of experimental work on the performance of a modern prototype automatic titrimeter for general analytical work. The instrument consists of a burette system controlled by a solenoid-operated valve so that the burette can flow at its max. rate to a pre-determined stage near the end-point, the remainder of the titration then taking place at a slow drop rate until, at the end-point, the flow is automatically stopped. Examples are given of the application of the instrument to the titration of H_2SO_4, H_3PO_4 and H_3BO_3 with standard alkali, halides with standard $AgNO_3$ and of I with $Na_2S_2O_3$. A further application is the maintenance of a definite pH during titration of F′ with $Th(NO_3)_4$ soln. A. O. JONES

818. **A new cell design for precision conductimetry.** J. C. Nichol and R. M. Fuoss (*J. Phys. Chem.*, 1954, **58** [9], 696–699).—The construction of a conductance cell for use with non-aqueous solutions at low concn. is described. The cell design permits interchange of electrodes in a vessel with a standard taper opening; the design does not involve stray electrical paths. Only one soft-glass platinum bead seal is necessary. Frequency variation is significantly less than that obtained with other cells. Resistances up to 50,000 ohms can be measured with a precision of 0·02 per cent. The plot of resistance against the reciprocal of the square root of the frequency (f) is nearly linear at low resistances, but curvature increases with cell resistance. Resistance is accurately a quadratic in $f^{-\frac{1}{2}}$; a numerical method of extrapolating results to infinite frequency, to eliminate polarisation, is presented.

R. J. MAGEE

819. **[Dropping-electrode] polarographs [of high sensitivity].** Nat. Research Development Corp. (inventor: G. C. Barker) (Brit. Pat. 709,826. Date Appl. 8.9.51).—A varying voltage of square wave form (amplitude 5 to 100 mV, frequency $\simeq$ 250 c.p.s.) is imposed on the usual d.c. voltage of the polarographic cell. During the rise and fall of the superimposed voltage, current in the cell circuit rises and falls rapidly, owing to the high double-layer capacity of the dropping-mercury electrode, and then decays exponentially to a "decay value" dependent on the cell diffusion current. The difference between the decay values is a measure of the difference in the diffusion current produced by the superimposed voltage; the magnitude of this change is governed by the applied d.c. potential and the ions present in the solution. Means are provided for monitoring the decay values of the current towards the end of each half-cycle of the square wave and a voltage proportional to the differences between consecutive decay values is impressed upon a signal integrator and passes thence to a recorder. J. M. JACOBS

820. **A new form of dropping-mercury electrode.** J. Smoler (*Coll. Czech. Chem. Commun.*, 1954, **19** [2], 238–240).—The usual type of dropping-mercury electrode used in polarography is modified so that its end is bent into a horizontal position. The mercury issuing from the capillary then forms a drop of minimum size with maximum stability. The dropping-time is reduced and this minimises galvanometer oscillations. The relationships between the diffusion current and the concentration, and between the diffusion current and the height of the mercury reservoir, are not affected by the modification. [This is a translation into Russian of a paper that was first published in *Chem. Listy*, 1953, **47**, 1667.] E. HAYES

821. **Coaxial trochotron for pulse counting.** O. Sternbeck (*Sci. Tools*, 1954, **1**, 11–13).—The coaxial trochotron RYG 10 is a high-vacuum tube with an axial oxide-coated cathode surrounded by ten control electrodes shaped to form boxes between them. The electron beam can be guided into any one of the boxes and retained in it until moved to the next one. The tube has a fluorescent screen, which indicates the position of the beam. Its incorporation in a decade counter is described.

A. M. SPRATT

822. **Carbon-film electric hygrometer element.** W. J. Smith and N. J. Hoeflich (*Bull. Amer. Meteorol. Soc.*, 1954, **35**, 60–62).—A new type of electric hygrometer, the "carbon-film element," is described. Its response to humidity changes is by electronic rather than electrolytic conduction. The main features are high sensitivity to moisture changes, rapid response, absence of polarisation effect and a calibration independent of temp. between 0° and 40° C. Components of the carbon film, performance data and manufacturing techniques are described.

S.C.I. ABSTR.

See also Abstract 558.

Important Notice

The Editor will be grateful if readers will inform him of any fields of analytical chemistry or of any journals that, in their opinion, are not adequately covered by "Analytical Abstracts."

Transliterations

From January 1955 the system of transliteration of the Cyrillic letters of Russian and related languages recommended by the Royal Society in their publication entitled "The Transliteration of Russian, Serbian and Bulgarian for Bibliographical Purposes" (February, 1953. Price One Shilling) has been adopted in "Analytical Abstracts."

Names of journals and authors therefore differ, in some instances, from those used in Vol. 1 in which English translations were sometimes used. It is hoped that the adoption of this system will eliminate any confusion that may have been caused by the earlier practice.

ABBREVIATIONS

Certain abbreviations in everyday use are not included in the following list. When any doubt might arise from the use in the text of an abbreviation or symbol the word is printed in full.

alternating current	a.c.
ampere	amp.
Ångstrom unit	A
anhydrous	anhyd.
approximate, -ly	approx.
aqueous	aq.
atmospher-e, -ic	atm.
boiling-point	b.p.
British thermal unit	B.Th.U.
calorie (large)	kg-cal.
calorie (small)	g-cal.
centimetre	cm
coefficient	coeff.
concentrated	conc.
concentration	concn.
critical	crit.
crystalline	cryst.
crystallised	cryst.
cubic	cu.
current density	c.d.
cycles per second	c.p.s.
decompos -ing, -ition	(decomp.)
density	ρ
density, relative	d or wt. per ml
derivative	deriv.
dilute	dil.
direct current	d.c.
distilled	dist.
electromotive force	e.m.f.
electron-volt	eV
equivalent	equiv.
experiment	expt.
foot, feet	ft.
gram	g
gram-molecule	mole
half-wave potential	$E_{\frac{1}{2}}$
horse-power	h.p.
hour	hr.
hydrogen ion concentration	$[H^{\cdot}]$
hydrogen ion exponent	pH
inch	in.
infra-red	i.r.
insoluble	insol.
kilogram	kg
kilovolt	kV
kilowatt	kW
maxim -um, -a	max.
melting-point	m.p.
micro-curie	μC
microgram	μg
micro-litre	μl
micron	μ
milliampere	mA
milli-curie	mC
milligram	mg
millilitre	ml
millimetre	mm
millimicron	mμ
millivolt	mV
minimum	min.
minute (time)	min.
molar (concentration)	M
molecul -e, -ar	mol.
normal (concentration)	N
number	no.
observed	(obs.)
ounce	oz
part	pt.
patent	pat.
parts per million	p.p.m.
per cent. wt. in wt.	per cent. w/w
per cent. wt. in vol.	per cent. w/v
per cent. vol. in vol.	per cent. v/v
potential difference	p.d.
pound	lb
precipitate	ppt.
precipitated	pptd.
precipitating	pptg.
precipitation	pptn.
preparation	prep.
qualitative, -ly	qual.
quantitative, -ly	quant.
recrystallised	recryst.
refractive index	$n\lambda^t$
relative humidity	R.H.
revolutions per minute	r.p.m.
saponification value	sap. val.
saturated calomel electrode	S.C.E.
second (time)	sec.
soluble	sol.
solution	soln.
specific gravity	sp. gr.
specific rotation	$[\alpha]\lambda^t$
square centimetre	sq. cm
standard temperature and pressure	s.t.p.
temperature	temp.
ultra-violet	u.v.
vapour density	v.d.
vapour pressure	v.p.
volt	V
volume	vol.
watt	W
wavelength	λ
weight	wt.

In addition the following symbols are used—

greater than	$>$	less than	$<$
not greater than	$\ngtr$	not less than	$\nless$
is proportional to	$\propto$	of the order of, approximately	$\simeq$

The principal Pharmacopoeias are denoted by B.P., U.S.P., or D.A.B., together with the identifying numeral.

Radicles are represented by the usual symbols; positive ions have superscript dots and negative ions superscript dashes, *e.g.*, $Cu^{\cdot\cdot}$, $Al^{\cdot\cdot\cdot}$, Cl', SO_4''. Metals that exist in more than one valency state are represented by their symbols with appropriate superscript roman numerals, *e.g.*, ferric iron becomes Fe^{III} and cuprous copper Cu^{I}.

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